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UTILISATION OF WASTE PRODUCTS

T. KOLLER

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THE UTILISATION OF WASTE PRODUCTS

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THE UTILISATION OF WASTE PRODUCTS

A TREATISE ON THE RATIONAL UTILISATION, RECOVERY, AND TREATMENT OF WASTE PRODUCTS OF ALL KINDS

BY

DR. THEODOR KOLLER

NET BOOK.

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GENERAL

A. I. T.

PREFACE

It will call forth no contradiction if we say that even to-day a vast number of substances are laid on one side as useless and are then exposed to the gradual decay which no organic body can escape, since it already contains the inherent conditions for decay—substances which, by rational treatment, would frequently be capable of further utilisation. Not rarely would such apparently worthless bodies produce a product, or even a series of products, which would make the value of the waste matter almost equal to that of the original material.

To show the truth of what has just been said, and the general high importance of waste products in regard to re-utilisation and economic yield, I may point to the chemical industries. Nowhere is the number of such cases so striking, numerically as well as economically, as in these industries.

I may recall for a moment the rich treasure in coal-tar, and mention the many oils and ethers, which so frequently, thanks to modern chemistry, owe their origin to residues and apparently worthless waste.

More than ever in these days it is necessary to pay careful regard to the apparently valueless. Competition compels all round the most economical, and consequently the most rational, labour; and apart from proper management—economical execution, division of labour, and the replacement of manual labour by the exact machine-power—the means of prosperity for everyone is to be found in the greatest possible utilisation of all waste.

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In the second edition, which has become necessary, I have endeavoured to add all advances, all new and approved processes for utilising waste matters; antiquated and unsuccessful processes have been omitted, numerous new practical methods of utilisation have been introduced. Thus I hope that the work, in its new form, may be and remain a practical guide and counsellor to those engaged in technical and industrial work, in order that the apparently worthless may be transformed into new and valuable products.

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THE UTILISATION OF WASTE PRODUCTS

INTRODUCTION

THERE can be no doubt that a rational utilisation of the waste matters which are produced so frequently and abundantly in the manufactures and industries, is of extraordinary importance.

Whilst we not rarely find that some waste product is accumulating in such quantity as to injure and retard the continuous progress of a branch of industry, we also see, not rarely, that the rational treatment and utilisation of such waste products either increase very considerably the general profits of an industry or even form a separate and not inconsiderable source of gain.

The numerous papers, which have had as their subject the profitable utilisation of waste matters, are frequently only to be found scattered in the form of occasional communications through the vast, widespread technical literature, so that, under ordinary conditions, it would be impossible for most men engaged in technical pursuits to discover, from among the abundance of material, that which is especially interesting to them and the requirements of their avocation.

It was no easy matter to exhaust the abundant material and arrange it, excluding all that was doubtful and superfluous, in such a form that the practical man, whatever his calling, might extract what is really useful.

Since the present work is the only one in the German language, which for the first time considers the treatment of waste products in a detailed manner and from a practical standpoint, the omissions which may appear here and there should be criticised in a friendly and well-meaning spirit. A smooth and well-trodden path may be traversed without danger even by an unsteady wanderer, but where the road must be first marked out and the rough path with difficulty smoothed, progress is a constant battle with hindrances.

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CHAPTER I

THE WASTE OF TOWNS

Utilisation of Town Refuse.—The question of the removal and rational utilisation of the waste matter of towns has for more than two decades occupied the attention of interested industrial circles. At first the chief exertions were directed to extracting everything in any way useful from town refuse, and especially to converting the fæcal matter into manure; but the rapid development of the large towns compels the first attention to be paid to the removal of this material. Meanwhile the warning voice of hygiene had been raised, and severe epidemics had taught the importance and results to the health of the dwellers in large towns, especially in regard to the prevention of epidemic disease, of a clean soil as opposed to a soil saturated with decayed and corrupt matter.

Thus it was that the question of the utilisation of town waste, in obedience to necessity, came more and more to verge into, How can the refuse matter of towns be removed in a manner satisfactory to hygiene? The industrial utilisation of this refuse took quite a secondary place.

So many difficulties are encountered in the manufacture of fertilisers from fæcal matter, which is subjected to so many precise and emphatic sanitary regulations, that, especially since it requires considerable capital, it has been quite forced into the background. Large towns and cities become more and more favourable to the water-carriage of fæces. This is because the most prominent authorities in hygiene designate this method as the most suitable. They cast aside reflections on the loss to agriculture of the material, and also remove doubts as to the advisability of the method by advancing the proved automatic purification of rivers.

The removal and utilisation of that waste of towns which may be described as household refuse, or "dust," is a different matter. In this case also a process of complete and innocuous destruction is to be regarded as the ideal. From the politico-economical point of view, and for the sake of the profit, it appears both desirable and justifiable to separate material of any value from the dust. The final solution of the problem will lie in the combination of the two endeavours: to extract from the dust what is still of value and convert it into useful product, and to destroy completely what is valueless.

In England dust was, and is, frequently burned. The combustion takes place in special furnaces, called "destructors," according to the system of Freyer. Such a furnace burns all the refuse of a town, both dry and wet. It is said that, by means of the destructor, it is possible to burn dust to which a large quantity of inorganic, incombustible matter has been added. The furnace stands in a brick building, and is fed from above. To the platform of the building leads a stage, by which the dust-carts are brought up; they then deliver directly over the opening of the furnace. The dust gradually descends. It is once for all lighted by means of a small coal fire, and afterwards burns continuously. It slowly falls farther and farther, finally reaching the part of the furnace at which the heat is most intense. The burnt residue passes through a grate into an ashbox, from which it can be easily removed. The installation consists of several furnaces in juxtaposition—in most towns six, but from eight to twelve are also found. From the hygienic point of view, it is important to know whether the gases given off in the process of combustion are harmful to the neighbourhood. It has been found that the combustion is practically complete; however, in order to remove any carbon monoxide which may be present, a smoke-consumer is attached to the destructor. The temperature in the hottest part of the furnace cannot be measured; in the main flues it is about 300–400° C. An erection of this kind requires no great space.

In the immediate neighbourhood of London there are seven or eight destructors, some of which are within London, and closely surrounded by houses. In Whitechapel, for

example, the destructor is only sixteen feet removed from the wall of the nearest house, without injury to the inhabitants. Destructors are in operation in at least twenty-four English towns, and some have been in use for more than fifteen years. A furnace of this kind burns per week (Sundays excepted) 24 – 35 tons — *i.e.* about 30,000 kilos. The cost of the process varies: in England it is estimated that it costs about one shilling to burn one ton of dust; but in isolated cases the cost is reduced to about fivepence per ton. The products of the combustion have many technical uses. Mortar and stone are made from them; also, the heat which is given out in the combustion of the dust is still further utilised, as for driving compressed air installations, for electric lighting, etc.

In this connection, it is to be remarked that the dust of English towns is quite different in composition from that of continental towns, since in England coal is much more extensively used for heating private houses than in Germany.

Later, in 1894, further progress was made in the construction of destructors. The system is considered the best in which the destructor is composed of a group of furnaces in the form of cells, generally six in number, built of fire-brick, and covered by an arch, which forms the top of the reverberatory furnace. The dust is emptied into an immense hopper at the top, from which it passes, after being rapidly dried, on to grates, similar to those of boilers, in the lower part of the apparatus, where it is burnt. About every twenty minutes a fresh charge is let down and burnt. On the average, each cell of the destructor, in which the fire is never extinguished, burns from 30–35 tons of dust per week. The ash and clinker from the furnaces are ground, and mixed with one-third of their volume of hydraulic lime, thus forming a blackish cement, not of very agreeable appearance, but selling at eight shillings per ton, and used in buildings for staircases, and especially for drains. The specially valuable part of the process lies, however, in the utilisation of the heat in the production of steam. With a good apparatus, each cell of the destructor gives ten horse-power, which is obtained from a boiler immediately attached to the destructor. In twenty

English towns the steam produced in this manner is used for disinfecting clothing and bedding. At Hastings the steam produced in this manner is utilised in pumping sea-water, which is used for flushing the public urinals and sewers, and for watering the streets. At Southampton the steam produced by the destructor compresses air, by which the sewage is raised and carried away; at the same time, it produces energy for an electric lighting installation. In other places this power is used for driving chaff-cutters, for producing mortar and artificial manures, and for driving grinding and mixing machinery.

The importance of the removal and utilisation of dust at the present day, in view of the rapid growth of the great towns, is evident from the fact that London, for example, has to remove not less than a thousand tons daily. The experience of Hamburg is interesting. After the cholera epidemic an attempt was made to burn the refuse, and, following the example of London, a furnace for dust was erected. The experiment was favourable under the conditions then prevailing; the dust burnt alone, and only a certain quantity of coal was required to start the combustion. Later, the dust burnt alone, but produced a rough and apparently valueless clinker. In Berlin, also, a destructor was erected. The experiences of London and Hamburg were utilised: English workmen were engaged to build and work the furnace. Success was absolutely wanting, for there remained an incombustible residue amounting to sixty per cent. Thus, of 100 cwt. which were brought to, and had to pass through, the furnace, and which could only be kept burning by a great addition of coal, 60 cwt. had again to be taken away. The municipal authorities were therefore compelled definitely to abandon the experiment.

In the next place, private industry stepped into the field. Rather more than a year ago a quite new method of disposal came into prominence, known as the "Budapest process." It requires large-scale arrangements for sorting the dust, such as were installed at Budapest, combined with the burning of the combustible portions. An endless band conveys the dust; on both sides stand children to sort the rubbish. One has to

pick out green glass, another white glass, a third rags, a fourth bones, all of which are thrown into baskets behind the pickers. Then the contents of the particular baskets are collected, and everything still usable utilised.

In Berlin this method was rejected for sanitary reasons. Dr. Weil, an authority on sanitation, gave a lecture to the Society for Public Health, in which he stated that this so-called Budapest system, which was advanced in Berlin as quite novel, had already been forbidden in London by the law in 1892. The advocate of the system could not answer Dr. Weil, whose statement still remains unrefuted, so that it may be accepted as correct.

The method employed for utilising the household refuse of Munich at the large establishment at Puchheim, on the railway from Munich to Buchloe, is similar to the Budapest process. I am obliged to the managers of this large establishment, now the third of the kind in Germany, for a complete insight into the method.

Every day two special trains, of thirty to forty waggons each, arrive in Puchheim from Munich. Each waggon contains four large trucks known as "harritsches." The refuse brought by these trains amounts to 450–500 tons daily, and is completely treated in ten working hours. The cost of railway carriage is about £18 per day, and amounts to over £5000 per annum.

The "harritsches" are taken up by a lift from the waggons, they are emptied by the bottoms dropping out, and are then brought back. The contents of each truck are raked by a workman through a shoot, and then sieved. The fine sievings are used as manure, for which purpose they are in demand, since they contain no small amount of nutritive matter. The price fixed by the management must be regarded as extremely low. The coarse residue then passes on to an endless band, from which, as in the Budapest process, it is sorted by women. The sorting produces a tolerable amount of valuable material. Especially considerable are the amounts of glass, rags, bones, and paper; even hare-skins are found in the season. Old shoes are also plentiful. About 500–600 bottles are collected every day. From the sorting band the

residue passes into a sieving drum, which has meshes of various widths. From the sieve the finer material, which is used as manure, is carried away by a screw conveyer.

The utilisation of the refuse is here carried out in the most complete and rational fashion. Unbroken glass bottles are washed and cleaned by an iron bottle-washing machine; they then again reach the brewer, wine merchant, etc. Broken glass is also washed in an iron drum; it then goes to the glass-works. The countless rags are beaten in a special rag-beater, by which they are opened out and freed from dust. They are then dried, disinfected, and sorted according to the fibre. The fine dust is mixed with phosphoric acid, saltpetre, and dried blood in a special mixer. The compound is then filled into sacks for use as a fertiliser. Old iron, which is also found in considerable quantity in dust, is converted into green vitriol. Old shoes are used for obtaining ground leather, which is mixed with the fertiliser. Large quantities of tinplate boxes may be treated in order to obtain the tin.

The works are plentifully supplied with steam-power, which is used for all the mechanical operations, and also carries out the electric lighting of all the rooms. There are about 3000 metres (nearly two miles) of railway track in the works.

The residue, which absolutely cannot be utilised, is carried away on rails in small trucks, three of which are drawn by an ox; it has hitherto been used for levelling the surrounding moor, but will shortly be destroyed by burning; the necessary furnaces are already erected.

From the sanitary point of view, the provision of lofty and airy working rooms, and of various ventilating fans, has satisfied the demands of health.

The process of utilising household refuse at Puchheim, which is allied to the Budapest method, is doubtless more rational, and may at the present time be described not only as the most important of the kind, but also as the best equipped and best managed.

The dust-melting process, based on the patent of Wegener, differs from the above. The dust is fused at a temperature of about 2000° C. No sorting takes place, but

the dust, as it is removed from the houses, is emptied into the furnace, which it leaves as a fluid glassy slag, which at once hardens when it has left the furnace.

This process might certainly satisfy all the requirements of sanitation. The question as to the cost remains to be solved. It has, however, been shown that the dust is fusible, and leaves nothing but the above-mentioned slag. The cost ought not to be unsurmountable, for the householder has already to bear the expense of removing the dust and of its disposal. On the ground of cost, it is necessary to utilise the heat of the furnace gases, which reach the flue at 1250° C. Anyone acquainted with the subject knows that this heat can be utilised. It is only necessary to measure the temperature in order to be able to show how many horse-power are available. This energy, according to measurements in fusing the total refuse of Berlin, amounts to 2800 horse-power per hour. There is no appreciable difficulty in utilising it; it is, on the contrary, an important factor in calculating the cost of the process.

Various experiments have been made with a view to utilising the slags, which constitute the residue in this process of fusing dust. It appears to be best to temper the slag, which flows out, by gradual cooling, so that it is not excessively brittle. The slag has also been powdered under stamps, and used as a substitute for emery. According to previous experiments, its hardness is 9°, the same as that of flint. It is also stated that this slag binds completely with cement. Since the slag is quite fireproof, it may well find employment in building, in particular for foundations. Taken on the whole, it is maintained that, according to previous experience, the fusion of dust would not cost more than the Berlin municipality now charge at their stations to the contractors for unloading—10 pfennigs per centner (about 1¼d. per cwt.). If the excess of heat were completely utilised, this cost should be considerably diminished. It should be observed that it is proposed to dispose of the residues or slag, which, as compared with the 60 per cent. of Hamburg, only amount to 12 per cent., to glassworks, where they would be ground and mixed with the glass charge.

In regard to the fuel, coal-dust firing is employed, and the intense heat can only be obtained in this way. A grate is consequently unnecessary, the coal dust is automatically thrown down from above by a special appliance. The dust at once takes fire at the high temperature and forms gases. The whole furnace is a mass of flame. No hearth is seen, but only burning gases, which fuse the dust. They then go, when this work is done, under the furnace to the flue, and should there be utilised.

Above the furnace is a large opening for charging, provided with a double cover, which fits air-tight and prevents loss of heat. The upper cover is taken off, the lower remaining closed, a box of dust is then emptied on the latter, almost filling the space between the two. The upper lid is closed, and the lower lid, the valves of which open downwards, opened by means of a mechanism, so that the dust falls by its own weight into a pipe leading directly to the furnace. In this retort the combustion begins, the temperature of 800° C. dries the dust, all combustible constituents are gasified, and thus produce fuel for the fusion of the incombustible portion. From the retort the unburnt dust, containing ash, crockery, iron, tinplate, etc., falls gradually on to a firebridge, where the temperature of 1200–1400° C. softens some of the materials and causes the others to sinter. Subsequently the 2000° C. of the furnace liquefies everything present in the dust, and the liquid flows out continuously. The furnace is built of fireclay.

CHAPTER II

AMMONIA AND SAL-AMMONIAC

Rational Methods for obtaining these Substances by treating Residues and Waste

Preparation of Ammonia from Guano.—In 1841, Young took out a patent for obtaining ammonia from guano (Muspratt's *Chemie*, 3rd ed., 1874, vol. i.). Vertical cylindrical retorts are filled with two parts of guano and one part of slaked lime or another caustic alkali. The two substances are mixed with a stirrer, then gently heated, and the temperature slowly raised to a red heat. Through the combined action of heat and the alkali, the whole of the nitrogen in the guano is converted into ammonia. Thus not only the ammonium salts already present, but also the nitrogen of the organic compounds, always present in guano, are obtained in the form of ammonia.

Preparation of Ammonia Solution from the Ammoniacal Liquor of the Gasworks.—The patented process of Watson was carried out in the following manner (Muspratt's *Chemie*, 3rd ed., 1874, vol. i.):—The crude ammoniacal liquor, together with a quantity of lime, which varies according to the quality of the liquor, is brought into a roomy retort or a boiler connected with a cooling apparatus. On warming, the gas is soon driven off, and is absorbed by the water in the receiver. On longer heating, much water distils over with the ammonia. As soon as this is observed, the receiver is changed and the later portions of the liquid caught separately, since the last portions are not alkaline, or only very slightly. This portion is brought back into the retort in a following operation, in order that it may be concentrated by a second distillation. The first portion distilled over is also not yet

sufficiently pure; it generally contains certain oily substances which can be mechanically separated, and, in addition, a tolerably considerable excess of water; it must therefore be subjected to a second distillation alone. The ammonia gas is, as before, caught in a little water, and the receiver changed when too much water goes over with it. Thus a solution of ammonia is obtained, which is sufficiently pure for almost all technical operations. The second portion of the distillate is reserved for the next operation, when it is again brought into the still.

Concerning this process, Stohmann (Muspratt's *Chemie*, 3rd ed., 1874, vol. i.) remarks with justice that it is quite unpractical, since several distillations are required in order to produce a commercial product. This may be avoided, he continues, by a methodical distillation, in which, during the operation itself, the quantity of ammonia contained in the vapours is increased, and the foreign constituents separated. The apparatus to be used for this purpose is an improved spirit still, by means of which a strong alcohol, free from fusel oil, may be obtained immediately from the mash liquor.[1]

The Dry Distillation of Bones produces a liquid which contains a large quantity of ammonium carbonate. Laming obtained ammonia solution from this liquid by first adding calcium chloride. Insoluble calcium carbonate and ammonium chloride (sal-ammoniac) are then formed. The liquid was then separated from the precipitate and boiled for an hour. All the volatile and gaseous impurities were thus removed. As soon as the liquid had cooled, it was mixed with a sufficient quantity of ferric hydroxide, by which all the sulphur compounds were converted into insoluble sulphide of iron. Milk of lime was then added, in order to set the ammonia free, and the liquid finally distilled.

L. l'Hote (*Compt. rend.*, 76, p. 1085; *Dingler's Journ.*, 209, 1873) utilised the waste of wool, hides, leather, horn, feathers, and sponges, which contains 6–15 per cent. of

[1] Newton used for this purpose, according to a patent in 1841, the still devised by Coffey, by means of which ammonia solution of any required strength, even to the strongest, can be obtained. Rose has also constructed an apparatus for the distillation of gas liquor. Compare Fleck, *Fabrikation chemischer Producte aus thierischen Abfällen*, p. 150; Knapp, *Chem. Technologie*, 3rd ed. vols. i. and ii. p. 534; Muspratt's *Chemie*, 3rd ed. vol. i. p. 648.

nitrogen, in order **to obtain ammonium sulphate**. When these waste matters are treated with a solution of caustic soda in from nine to ten times its weight of water, which should be done in the cold or at a gentle heat in order to avoid loss of ammonia, some are dissolved, whilst some entirely lose coherence. The slimy liquid so obtained is mixed with slaked lime to a paste, which is brought into an iron retort, connected to receivers, in which is chamber sulphuric acid. The mass is then distilled at the lowest possible temperature, in order to prevent the dissociation of the ammonia; when the evolution of gas has ceased, the retorts are heated to a red heat. After the operation there is found in the retorts a white powdery residue, consisting only of sodium carbonate and quicklime. If this residue be treated with water, caustic soda is again formed, and can be used for the next operation. The ammonium sulphate obtained is coloured; it may be purified by recrystallisation.

The following process (*Ber. d. d. chem. Ges.*, 1873, 1553) was patented in England by W. M. Brown, for J. H. Elvert, of Geneva, and J. J. M. Pack, of Basle, for **the preparation of pure ammonia from gas liquor**:—The liquor is mixed in a closed vessel—cylinder or boiler—with the proper quantity of lime, drawn off into a second vessel, and there heated. The vapours and gases evolved are conducted to the bottom of the first vessel, which has meanwhile been recharged with gas liquor and lime, and thence, now containing more ammonia, to a condensing chamber, from which the more readily condensible constituents flow back through a connecting pipe to the last-mentioned vessel (No. 1), whilst the more volatile constituents pass through a cooling coil into a second condensing chamber, where hydrocarbons, sal-ammoniac, and a small quantity of free ammonia are retained. The purified vapours, which are not yet condensed, are conducted through several vertical pipes filled with charcoal to receivers containing distilled water, which are changed as they become saturated. As soon as all the ammonia has been driven out of still No. 2, it is emptied, filled with the contents of No. 1, the latter charged as before, and the operation recommenced. When treated in this

manner, 1000 litres of crude liquor at 3° B. give, in the course of from four to five hours, 100–110 kilos. of commercially pure ammonia of 22° B.

The apparatus hitherto used for the distillation of the ammoniacal liquor of gasworks either consists of a still, in which the liquor is distilled alone or with lime, or two vessels are used, one of which serves to heat the charge, and the other as the still. If no lime were added, only the ammonia combined with volatile acids would be obtained; in the other case the lime introduced into the apparatus caused danger of burning the still, a danger only imperfectly removed by the introduction of a stirrer.

This evil is removed by an arrangement invented by H. Grüneberg, of Kalk, near Deutz (*Polyt. Journ.*, vol. ccxxix., 1878; Ger. Pat. 351, of July 13, 1877), which enables lime to be used without a mechanical stirrer, and thus brings about the saving of the whole of the ammonia, including that combined with hydrochloric and sulphuric acids, without causing injury to the apparatus.

The fresh ammoniacal liquor comes from the tank into a boiler 7–8 metres long (23–26 feet), where it is heated; it then passes into the second boiler, heated by direct fire, thence to the third boiler, which is not in contact with the fire gases, where it is treated with lime to decompose the non-volatile ammonia compounds; finally, after exhaustion, it is run off. In regular working, the second boiler is heated; the fire gases which leave it surround the first boiler, and bring its contents to the boiling-point. The vapours evolved in the second boiler pass into the third through a pipe which has a rake-shaped end; in this boiler they drive out the ammonia set free by the milk of lime, which is introduced through a funnel. The ammonia, together with steam, passes through a pipe into the preliminary heater, where it takes up the volatile ammonia compounds, and, with these, passes through pipes into covered leaden boxes, filled with 50 per cent. sulphuric acid. The gases evolved here, principally sulphuretted hydrogen and carbonic acid, are taken through a flue to the fireplace of the second boiler, and so made harmless.

When the acid in the saturators is neutralised, the liquor

obtained is allowed to cool and the crystallised salt collected on drainers, whilst the proper quantity of sulphuric acid is added to the mother liquor and the mixture treated with ammonia vapours, so that a separate evaporation is not required.

If sal-ammoniac is to be made, the leaden boxes are replaced by stone tanks filled with hydrochloric acid; in this case evaporation of the liquid cannot be avoided.

This apparatus has already been introduced into the ammonia works at Cologne, Hamburg, Stettin, and Leipzig. It has been found that it is easy to work, that the necessary repairs are small, and that the gas liquor is completely utilised.

Sal-ammoniac from Gas Liquor.—The liquor is treated (Stohmann, Muspratt's *Chemie*, 3rd ed. vol. i.) either by distilling with a quantity of lime, proportioned according to the quantity of ammonium chloride or sulphate to be produced, and the evolved ammonia led into hydrochloric acid, or the gas liquor is immediately neutralised by ammonia. The finest product, almost chemically pure, is obtained by the former process.

In this process the liquor is heated with quicklime in a capacious still to boiling, the vapours taken through a coil cooled by water, in order to condense the greater quantity of the water, and the distillate allowed to flow into a tank containing hydrochloric acid. This tank is a wooden box lined with thick sheet lead, and closed by a cover. A pipe takes the unabsorbed gases—sulphuretted hydrogen, etc.—under the fire-grate of the still, where they are burnt and then pass into the chimney. The end of the coil dips into the acid, so that the ammonia, which is only partially condensed in the coil, may be entirely absorbed.

After about one-tenth of the liquid in the still has evaporated, all the ammonia appears to be driven out, a point which is recognised when a red litmus paper, held in the steam issuing from a test-cock, is no longer, or only slightly, turned blue. This tap is then opened and the still emptied, the air entering through the tap and thus preventing the sal-ammoniac solution from rushing back.

The ammonia gas is allowed to pass into the acid until the latter is just neutralised, when the salt solution is drawn off and the receiver charged with fresh acid. Generally the sal-ammoniac solution is so concentrated that it need not be much evaporated in order to attain the strength requisite for crystallisation. If it is then allowed to cool, a fine crop of crystals, almost quite white in colour and completely free from tarry matters, is obtained.

The distillation can be more conveniently conducted if two boilers are connected together by pipes and cocks in such a manner that the vapours from each can be passed into the other. The first boiler is then first heated by direct fire; the latent heat of the steam brings the liquor in the second to boiling and volatilises the greater part of the ammonia, so that the ammonia from both quantities of liquid passes in the vapours from the second boiler into the acid. As soon as the first boiler is exhausted, its contents are run off; it is then refilled and fire made under the second boiler, in order to drive steam from it into the first and the ammonia thence into the acid, until the second vessel is exhausted, when it is again filled. Great economy in fuel is thus obtained, since double the quantity of ammonia can be obtained by using almost the same amount of fuel which is required for the distillation of the liquid in one boiler. There is also the advantage that far less steam passes over with the ammonia gas, and thus a much more concentrated sal-ammoniac solution is obtained.

Utilisation of the Residue of the Caustic Alkali Manufacture.—The caustic soda liquor (and caustic potash similarly) is made from 10 parts of calcined soda, 100 parts of water, and 20 parts of fresh slaked lime, by dissolving the soda by boiling, and then gradually adding the powdered and sieved lime. The boiling must be continued until there is no more sodium carbonate in the solution, the carbonic acid having united with the lime to form carbonate of lime, whilst caustic soda remains in solution. After cooling, the clear liquor is carefully drawn off and brought into closed bottles or carboys, so that it may not absorb carbonic acid from the air. The paste remaining in the boiler is then brought into linen

strainers, previously moistened with water, where the caustic liquor drains off, leaving behind the carbonate of lime. As a rule, the residue in the strainer is washed with warm water, when a somewhat weak caustic liquor is obtained, which can be added to a fresh operation, *i.e.* when fresh caustic liquor is boiled. The washed paste is then brought into wooden boxes, which are perforated at the bottom with very fine holes, over which coarse linen is laid. Here more liquor drains away. The stiff mass is moulded in moulds similar to those used in brickmaking. The bricks are dried on boards in the air; the dried bricks are then very finely ground and sieved in order to remove all impurities. The fine powder may then be used in various manufactures (*Neueste Erfindungen und Erfahrungen*, 1896):—

In the preparation of an artificial chalk, for which some binding medium is requisite, the mass is made plastic with an addition of water-glass as binding agent, then pressed and slowly dried. The mass thus becomes as hard and firm as natural chalk; it can easily be cut up for use as writing chalk.

For the manufacture of a fertiliser for grass: the dried residue always contains a few per cent. of alkali, which cannot readily be removed by washing. In combination with other fertilisers this is very useful, especially when it is mixed with sieved wood ashes, bone meal, ground excrement or dried blood in certain proportions. The following are very good proportions:—

Fifty parts of the ground alkali residue, 25 parts sieved wood ashes, 20 parts bone meal, 20 parts excrement, 10 parts dried blood.

Another composition: 50 parts ground alkali residue, 50 parts bone meal, 30 parts ground excrement, 30 parts sieved wood ashes.

And another: 60 parts ground alkali residue, 60 parts sieved wood ashes, 30 parts ground kainite, 40 parts ground excrement.

These are all well mixed, sieved, and packed into casks, which must be well closed.

Utilisation of the alkali residue in making polishing powder and paste. The following recipes are given:—

Polishing powder: Finely powdered residue (containing soda), 50 parts; finely powdered rouge, 20 parts; finely powdered magnesia, 10 parts; finely powdered glass, 20 parts. These substances are very thoroughly ground together and the mixture sieved.

Polishing paste: Finely powdered residue (containing soda), 60 parts; kieselguhr, well sieved, 40 parts; rouge, 20 parts; glass, finely powdered, 20 parts; magnesia, 10 parts. These substances are well mixed, and then well ground with oleïne to a paste on a paint mill.

CHAPTER III

RESIDUES IN THE MANUFACTURE OF ANILINE DYES

Utilisation of the Residues.—In the manufacture of magenta resinous residues are produced, in which is contained, in addition to organic matter, almost the whole of the arsenic originally employed.

According to Bersch, this arsenic may be regained in various ways. The arsenious and arsenic acids in the residues are first brought into solution by boiling with hydrochloric acid, and extracting the undissolved residue with water. The two solutions are mixed and neutralised with soda, when a dark green precipitate is produced. The liquid filtered from the precipitate, which is now a solution of sodium arsenite and arsenate, is mixed with milk of lime, which precipitates calcium arsenate. The precipitate is decomposed, in vats lined with lead, by sulphuric acid, to which a little nitric acid has been added. The solution of arsenic acid is drawn off from the deposited gypsum and again used, after concentration, for the oxidation of aniline oil.

In order to work up the mother liquors of the magenta manufacture for arsenic acid, the acids of arsenic are precipitated by lime, the precipitate washed and dried, and the arsenic reduced by ignition with coal. The arsenic vapours evolved are allowed to come in contact with hot air, by which they are burnt to arsenious acid, which is condensed, and again oxidised by nitric acid to arsenic acid. This process is, however, only suitable if concentrated liquors are in question. Weak liquors should be evaporated in shallow pans, which can be heated by the flue gases from other apparatus; after this concentration they may be treated by the former process.

Another method, which (according to Bersch) is suitable for works with very cheap fuel, and to working on a very large scale, consists in mixing the very concentrated solution of sodium arsenate with powdered chalk and lignite, drying, and heating the mass in a furnace. The vapours of the (reduced) arsenic are allowed, by contact with air, to burn to arsenic trioxide, which is condensed and deposited in long flues. The residue in the furnace consists of soda mixed with calcium carbonate; it is extracted with water, and thus both substances may be again used in succeeding operations for recovering arsenic.

In order to obtain magenta from the mother liquors left after the crystallisation of the dyestuff, soda solution is added, which separates the magenta. From the residual liquid the arsenic is regained by one of the methods described.

If the mother liquors are to be worked up systematically, shallow leaden pans must be employed, which are closed and provided with vapour pipes leading into a chimney with a good draught. When possible, the pans are heated by the flue gases from the vessel in which aniline is transformed into magenta. The very concentrated mother liquors yield a considerable quantity of sodium arsenate, and the liquid still remaining may, in order to obtain the rest of the arsenic, be precipitated with lime, and the precipitate converted into arsenic acid, as previously described.

By systematic treatment of the impure mother liquors from the manufacture of magenta, several firms have succeeded in producing certain definite shades of reddish-brown and brownish-red, passing over to the deepest brown. It is of the first importance always to produce these of the same nature; so that the same shade of colour may always be produced by a dye which has once been known in commerce under a certain name. This object may be attained, according to Bersch, by always working by one and the same process in the manufacture of magenta, and by always treating the mother liquors by the same process of evaporation.

Treatment of the Residues produced in the Manufacture of Aniline Red.—The methods with arsenic acid

and mercuric nitrate, as also Coupier's process with nitrobenzene, iron, and hydrochloric acid, give only a yield of 30–40 per cent. of the aniline employed in the manufacture as saleable dye. The Actiengesellschaft für Anilin-Fabrikation, of Berlin, has now found, according to a patented process, that the air-dried residues give, on dry distillation from horizontal retorts, a distillate which, together with water and ammonia, also contains aniline, toluidine, xylidine, and their homologues, which can again be directly employed in the manufacture of aniline red; and, in addition, a high-boiling oil, which consists principally of diphenylamine, and contains also naphthylamine and acridine. The residual coke is used as fuel.

CHAPTER IV

AMBER WASTE

Utilisation of the Waste.—Attempts have been made to weld amber cuttings together by heat, but never with great success; the mass obtained has lost somewhat in beauty of colour by being turned brown by the heat. A better process is the following:—After the cuttings have been sorted according to size, they are treated for several hours by solvents such as carbon bisulphide, ether, etc. A plastic mass is obtained, which is spread out on a table so that the excess of solvent may evaporate, when it is compressed under great pressure into the requisite moulds.

In certain cases it would be advisable in this process to apply a gentle heat in order better to unite the isolated parts, and to press them better into the moulds.

The pieces obtained, which have already the general shape of their final form, are further treated in the ordinary manner and then polished.

Whilst the lumps of amber welded together by the old process are very brittle, those obtained by the process just described are characterised by great elasticity. Holes may be bored in them without fear of cracks or breaks, which often occur in the pieces amalgamated by heat alone.

In order to obtain articles of a cloudy or speckled appearance, small pieces of hard amber are mixed with the plastic mass; these pieces, when they differ in colour, give the article a novel character.

CHAPTER V

BREWERS' WASTE

Utilisation of Malt "Combs."—These are an excellent food for cattle.

Utilisation of Brewers' Grains.—They are used as food for cattle. They should be employed as fresh as possible, since lactic acid readily forms, which then later produces butyric acid, acetic acid, etc. This objectionable alteration of the grains is recognised by the loss of the pure malt odour, the smell becoming distinctly sour. In feeding with grains it is necessary to add chopped hay or straw in order to cause the cattle to masticate. For a short period—about fourteen days—the grains may be kept in tanks, in which they are weighted down with boards and stones, and covered with water to a depth of several inches. The tanks must be kept very clean, and when emptied, must be lime-washed until the coating does not turn grey—which is a sign that acid is present—but remains white. When kept longer, brewers' grains are deposited in a pit in the ground and covered by a thick layer of earth. The pits should be of such a size that the grains in each are used in eight, or, at the most, fourteen days. Fermenting grains are a valuable manure.

There are four methods of preserving grains to be considered. The first, drying in the kiln, gives very good results, but the process is too expensive. The second, which is coming into use in England, consists in expelling the moisture in centrifugals, into which hot steam is conducted; the grains then become pressed into solid cakes, which are recommended as a most excellent food for cattle. In another method, the grains, mixed with other suitable foods, such as bran, meal, peas, beans, etc., are mixed up to a paste, and baked into a kind of bread. The product is brown, porous and friable, smells

and tastes similarly to fresh rye bread, mixes with water, and may be given dry mixed with other chopped fodder, and also as a drink. Finally, storage, with an addition of common salt, in pits with cemented brick walls, is said to have been effective.

Utilisation of the Yeast.—Pressed yeast, commonly known in England as German yeast, is produced. H. Rüdinger gives the following account of its preparation:[1]—

After the termination of the main fermentation, the yeast which has separated in the fermenting vats is collected in a large tub, provided with a series of plugged holes in the side. The mass is left at rest for some hours, when the yeast settles, and, by opening the upper holes, a quantity of raw beer may be obtained, which is either put into a barrel or added to the fermenting vat in the next fermentation. After this beer has run off, the tub is filled up with clean water, the whole well stirred and allowed to deposit. After about an hour the liquid may again begin to run off. The upper holes are opened; the liquid, which is at first clear, is removed, but when it begins to be tolerably turbid, it is run through a sieve, on which a wet coarse linen cloth is spread. The linen retains the greater portion of the yeast; but after some time the pores of the cloth are so filled by it, that the liquid runs away very slowly. It is therefore necessary to have several sieves in readiness so that the operation may not be stopped.

The running-off of the liquid, by opening lower holes, is continued until the yeast begins to appear as a thick liquid, when the holes are again closed. The yeast on the linen is removed and returned to the tub, which is again filled with water, and the yeast treated exactly as before. This washing is continued so long as the yeast on the linen has a fairly strong bitter taste. In most cases three or four treatments of the yeast, with fresh quantities of water, are sufficient to remove the bitter taste to the required extent.

The washed yeast is now collected and freed from water, for which purpose a system of filters is arranged near the

[1] *Die Bierbrauerei und Malzextract-Fabrikation*, H. Rüdinger, published by A. Hartleben, Vienna, 1887.

yeast-tub. The uppermost part of this apparatus is a fine hair-sieve; below this are placed several frames, across which linen cloth is stretched, being hung at the four corners of the frame to wooden pegs by means of stitched-on loops. The turbid liquid coming from the top cloth leaves a portion of its yeast on the next, also on the third, and so on, finally leaving the last cloth with only a slight turbidity; it is received in a tub, where it deposits the remainder of the yeast as a fine, slimy residue, whilst the supernatant liquid is quite clear.

The pores of the filter cloths are soon so filled by the fine particles of the yeast that only a little liquid can pass through; frames in this condition are removed, allowed to drain spontaneously, and replaced by fresh frames. When finally all the liquid has been brought from the tub on to the filter frames, the latter are allowed to stand until no more liquid drips from them. The yeast now appears upon the cloths as a brownish-coloured soft mud, which is removed by spreading the cloths out flat, and scraping off the yeast with a blunt wooden blade.

The paste is now brought into a very strong cloth (press cloth), this is surrounded by a second cloth, and the whole brought under a screw press, where at first a gentle pressure—the liquid should not run away turbid—and then gradually a considerable pressure is applied. When once a considerable quantity of water has been pressed out of the yeast, the pressure may be allowed to rise tolerably high, so that the compressed yeast taken out of the press cloths is a mass which may be compared in consistency with new cheese.

In order to be able to keep the pressed yeast without alteration, it is advisable at once to divide it into rectangular blocks of definite weight (500, 250 grms., *i.e.* about 18 or 9 oz.), which are then wrapped in oiled paper or tinfoil, and kept in a cool place.

Utilisation of the Bottom Yeast.—If bottom yeast is to be converted into useful bakers' yeast, it must be washed. It is first run through a fine hair-sieve into a vat, which is somewhat deeper in front than behind. In the front wall of this vessel, in the middle stave, which may be somewhat

thicker than the others, in order not to lose strength, are bored holes at intervals of 5 cm. (2 inches) at the top, and at smaller intervals below; the holes are now closed with plugs. The yeast is stirred with water, to which a good eggspoonful of powdered ammonium carbonate is added per hectolitre of yeast (22 gallons). When the yeast has settled, the plugs are drawn one after the other, and the water allowed to run off. The yeast is now rarely white enough, so that the process must be repeated a second and third time. The second time less ammonium carbonate is added; the third time, none.

This process is occasionally also used for top fermentation yeast, in which case two additions of water, without the salt, are generally sufficient. When the yeast is white enough, it is filled into press bags and well pressed; it is also frequently kneaded with starch, partly to improve the colour, partly to give it more the nature of a so-called short or crumbly yeast.

Utilisation of the After-Worts.—These are advantageously employed, instead of water, for mashing the malt of the next mash, if that takes place immediately after the one from which the after-worts were obtained. If it would be necessary to wait only several hours, it is advisable to use the after-worts together with the wash-water of the grain for preparing malt vinegar. For the same purpose, *i.e.* vinegar, all other residual liquors—the last portions of the beer, which are turbid owing to the presence of yeast, etc. —may be employed.

For the last-mentioned purpose a wide but low vat, provided with a cover, is brought near the brewing vessel. The liquids enumerated above are brought into this vat. In order to commence the formation of vinegar, it is only necessary to introduce a few glasses of sour beer or some "mother of vinegar." After the liquid has stood for some hours in this vat, it may be drawn off into smaller vessels, placed in some situation at an ordinary room temperature. If the vinegar is found to be too weak, one-hundredth of its volume of distilled spirit is added, for which purpose the crude distillate may be used.

Utilisation of the Deposit in the Coolers. — This deposit may be used as fodder in conjunction with brewers' grains or distillery wash.

The Utilisation of Spent Hops as Cattle Food has been treated by O. Kellner.[1]

The spent hops (according to Kellner), on account of their composition, which corresponds to that of red clover hay of medium quality, have recently been much recommended for fodder. Since, on the one hand, this refuse is produced in large quantity and has hitherto been utilised to an inadequate extent, and, on the other hand, it was of great interest to learn the behaviour of such an extracted vegetable substance towards the digestive juices, Kellner undertook to ascertain the digestibility of spent hops by a direct nutritive experiment. The results showed that the digestive co-efficients of the constituents of hops, with the exception of that of the crude fat, are lower than have hitherto been found for any food directly tested for digestibility. Since the extraction in the brewing did not afford a sufficient explanation of this remarkable result, other conditions were included in the scope of the observations. It was then found that about 24 per cent. of the crude fibre is lignin, which, being in intimate admixture with the cellulose, considerably decreases the digestibility of the latter. Also in spent hops a portion of the proteïn is found in combination with tannic acid, which is known to hinder the action of the digestive juices. Only the crude fat had a normal digestive value. Since this constituent of hops is certainly composed of substances which have not the chemical constitution of ordinary fats, it may also be assumed that the digestibility of the hop fat is not equal to that of the ordinary fats.

Thus, in consequence of the low digestibility and the reluctance with which cattle eat spent hops, this substance cannot find any extensive use as fodder.

The residues in question are best employed in preparing compost, perhaps after the residual portions of the wort have been removed by water, to be further utilised in beer. Small

[1] *Deutsche landwirthschaftliche Presse*, 1879, No. 55, pp. 332, 333; Biedermann's *Centralbl. für Agriculturchemie*, 1879, No. 9.

additions of hops to the daily food are not to be rejected, since the appetite of the animal is increased thereby. The proposal of Pott, to add spent hops in place of the usual straw to the cakes of distillery and brewers' grains, appears to be worthy of attention, especially since the tannin in the hops has probably a preservative action. For the same reason spent hops might perhaps be used with advantage in storing sliced beet, etc.

The best method for treating *malt "combs"* for use as food or manure consists in grinding to a coarse meal, after well cleaning and removing dust. This substance must always be mixed with other foods; it is most suitable for improving a ration of chopped straw. By grinding, the "combs" are made more easily attacked, and hence more digestible. The same reason holds good when malt "combs" are to be used as a fertiliser. From experiments of A. Fericka, it appears that the differences in the vegetation obtained with different manures are very considerable, and that ground malt "combs" treated with sulphuric acid affords the best means of increasing the growth of grass. The process of treating the ground "combs" with sulphuric acid is as follows:—In a large tub, 5 cwt. of the ground "combs" are steamed, with the gradual addition of boiling water, until a paste is produced, which is left at rest for two hours, after which the whole mass swells up considerably, and it is then found that the meal has absorbed the whole of the water, no more remaining at the bottom of the tub. Then the mass is again scalded, this time with an addition of about 10 lb. of sulphuric acid to the boiling water. After cooling, the material may be at once used for manure; it is especially suitable for incorporation in poor composts.

CHAPTER VI

BLOOD AND SLAUGHTER-HOUSE REFUSE

Utilisation of Blood. — W. L. Palmer employs blood to obtain a plastic material. It is passed through a fine sieve, dried, pulverised, then mixed with 20 per cent. of bone meal and 10 per cent. of size, placed in the required moulds and subjected to a high pressure at 120° C. This application of blood, and also earlier uses, in which blood and sawdust were used in preparing plastic masses, are now of no technical importance, since cellulose can at the present day be employed much more successfully in making artificial wooden ornaments.

Treatment of Blood.—The great obstacle to the use of blood in manufacturing processes is its decomposition, which rapidly occurs. This obstacle may be combated in one way, by carrying out all the operations as rapidly as possible. Heinson Huch of Brunswick has also patented a simple process (*Neueste Erfindungen und Erfahrungen,* 1877) for protecting blood from decomposition and drying it. According to this process the blood, in the vessel in which it is caught from the slaughtered beast, is stirred as it flows in with about 3 per cent. of ground or finely powdered quicklime, *i.e.* about 450 grms. (one pound) for each bullock. After stirring for three to five minutes the mixture is allowed to stand. The lime rapidly settles to the bottom and the blood coagulates in a few minutes. It is then removed and placed in the sun in an airy position to dry, the drying being accelerated by stirring at intervals. In a short time the blood is dry; it remains free from smell and taste, and does not evolve ammonia. In the winter the drying must naturally be accomplished in a kiln, or a stove if the quantity is small. The ground or powdered lime comes in contact with all parts

of the blood during stirring; it unites with some of the water and sinks to the bottom without leaving perceptible traces in the blood, which only becomes somewhat more alkaline and dries more readily, without clotting fast together, as happens in evaporating. Since no smell at all is evolved, the blood retains the whole of its nitrogen, which is very important to its value as a fertiliser.

G. Thenius has also described the practical treatment of blood (*Neueste Erfindungen und Erfahrungen*, 1878). Bullock blood is that which is obtained in the largest quantity in slaughter-houses. In order to obtain it in a readily fluid condition, it is stirred with a stick for some time until the fibrin has separated in long threads, when the stirred blood is brought on to a fine hair-sieve and washed with water. The washed fibrin is placed on dry cloths, so that it may rapidly be freed from water, as otherwise it quickly decomposes. After this operation with dry cloths has been frequently repeated, the fibrin is placed on a hair-sieve and dried at 120° C. in an air-bath. It is to be observed that this fibrin still encloses some globulin, and cannot be regarded as pure. The globulin and also hæmoglobin, the colouring matter of blood, are always enclosed by the fibrin; they may be removed by long continued washing, in order to obtain pure fibrin.

The blood freed from fibrin does not decompose so rapidly and may in cold weather be kept for some time. The author treats the fluid blood mainly by two methods:—

1. By drying upon flat zinc sheets (previously oiled), the edges of which are turned up to prevent the fluid from running off, in drying-stoves, provided with shelves, and in which the temperature is maintained at 30–35° C. The dried blood, which is now obtained in thin transparent sheets, is used in sugar-works, for the sake of its albumin, for clarifying the juice. From 100 parts of the fluid blood about 21–22 parts of the dry substance are obtained; thus 78–78·5 per cent. of water has to be evaporated. The author has constructed a special furnace for this drying-stove, the arrangement of the flues in which has been found

to be very effective; a regular heat is maintained, and the expenditure of fuel is low (Fig. 1).

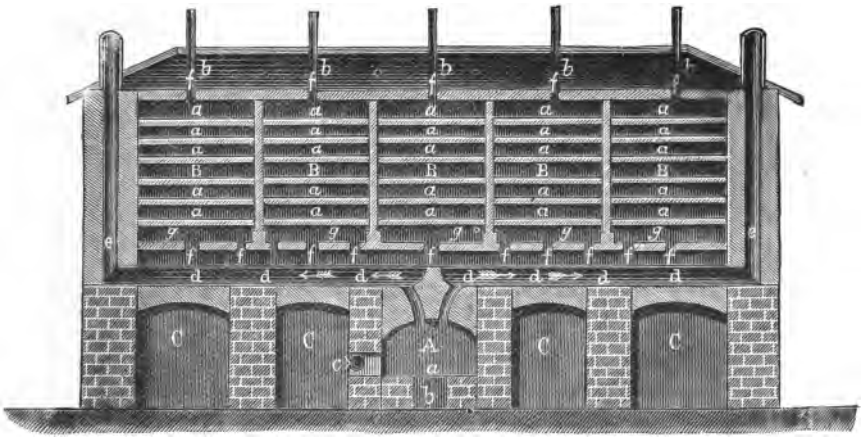

FIG. 1.—Vertical Section of the Furnace, with Drying-Stoves for Heated Air. *A*, Furnace; *a*, hearth; *b*, ashpit; *c*, door; *d*, flues; *e*, chimney; *f*, air passages; *g*, covering plates. *B*, Drying-Stove; *a*, shelves; *b*, pipes for the moist air. *C*, Workrooms.

2. By thickening the fluid blood in pots with continuous stirring, in order to obtain blood meal. The author has constructed for this purpose a special furnace (Fig. 2),

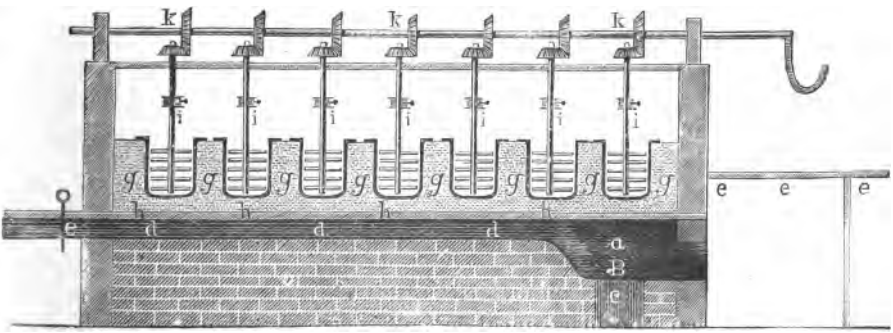

FIG. 2.—Longitudinal Section of the Furnace used in making Dry Blood Meal.

which has been very satisfactory. In the figure, *a* is the fireplace, *B* the grate, *c* the ashpit, *d* the flue; the chimney,

f, which receives the flue, is regulated by the damper, *e*. The flues are roofed with cast-iron plates, *h*, upon which is a sand bath, *g*, containing the iron vessels in which the stirrers, *i*, can be kept in motion from the shaft, *k*. Round the furnace is a platform, shown at *e*, to facilitate the removal of the pots and the actuation of the stirrers by a labourer.

The fluid blood is brought into the pots, which should only be half filled; they are then placed in the sand-bath and the stirrer *k*, *i*, put in position. The furnace is then heated and the stirrers set in motion, in order that the blood, which soon coagulates, may not stick to the walls of the pots and burn. When the stirring is continuous, the mass, which was at first pasty, is gradually converted into a pulverulent condition; it acquires thereby a dirty brown colour, which turns to reddish brown as the drying proceeds. The vapours evolved during this period have a peculiar odour and oxidise metals, such as copper, very rapidly. The workman in charge of this operation is almost intoxicated by breathing these vapours, especially when fresh blood is being treated. As soon as the pulverulent blood has become quite dry and no more vapours are evolved, the stirrers and pots are removed from the sand-bath, the pots emptied into sheet-iron vessels which can be well closed, and the contents allowed to cool. When quite cold the blood is dust-dry and can readily be ground. The average yield is 20·5–21·2 parts from 100 parts of fluid blood, so that, as a maximum, it may be assumed that 78·8 per cent. of water and gases are lost in evaporation. When the blood, whilst still somewhat moist and powdery, is filled into strong linen bags and subjected to a considerable pressure in a press, there is obtained a watery and fatty liquid, the fat of which solidifies at low temperatures and can be separated from the water. The quantitative yield of this liquid is 2·7–3 per cent. The aqueous solution contains the salts. The solid compressed residue is more adapted for storage than the blood meal, since it does not absorb so much gas or attract moisture. The dry blood may either be used for producing prussiate of potash, or for obtaining a very good charcoal containing nitrogen, and also as an admixture to artificial manures.

The following apparatus (Fig. 3) is used in making blood

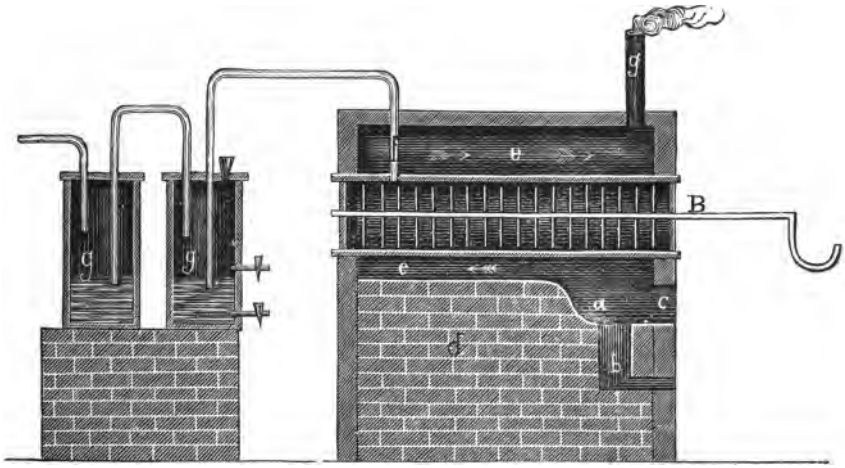

FIG. 3.—Longitudinal Section of a Blood Charcoal Furnace. *a*, hearth; *b*, ashpit; *c*, door; *e*, flue; *f*, exit-pipe; *g*, chimney.

charcoal. The dried and powdered blood should not more than half-fill the cylinder, since the mass swells on heating,

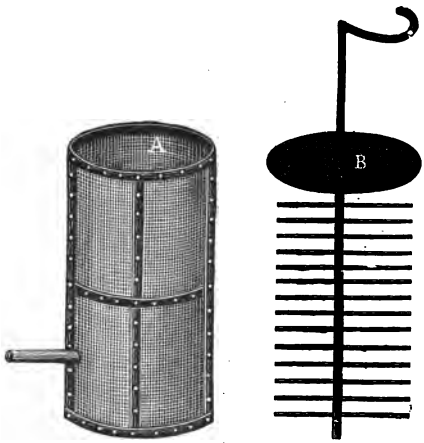

FIG. 4.—*A*, Cylinder for Blood Charcoal. *B*, Lid, together with Stirrer.

and the stirrer, *B*, must always be kept in motion. The

cylinder (Fig. 4) is made of cast-iron or thick sheet-iron and is constructed to remove from the furnace *d*. The stirrer, *B*, can also be removed together with the lid from the apparatus, in order to facilitate cleaning.

The vapours evolved, consisting chiefly of ammonia and empyreumatic oils, pass away by the pipe, *f*, to the receiver, *g*, which contains a leaden vessel, in which is dilute sulphuric acid. The ammonia combines with the acid to form ammonium sulphate, whilst the oily substances swim on the surface and can be utilised as Dippel's oil. Fresh dilute sulphuric acid must frequently be supplied to the receiver, *g*, after drawing off the saturated ammonium sulphate solution. As soon as no more vapours are evolved from the apparatus, it is removed from the furnace, allowed to cool for several hours with frequent stirring, then opened, and the fine blood charcoal brought into a vessel which can be tightly closed. This charcoal absorbs gases with such extraordinary rapidity that it is often so strongly heated as to become red hot. In medicine this preparation is known under the name of *carbo animalis*; it is principally used for dusting malignant wounds, which give off much bad gas and smell. When freshly burnt, it could be used for the disinfection of hospitals and similar buildings. If blood charcoal be ignited with potash it acquires a much greater decolorising power. On this account the charcoal is preferred in lumps, which contain many pores.

The most important technical use of dried blood is in the manufacture of potassium cyanide.

If dried pulverised blood or blood meal is allowed to lie exposed to the air, it soon gives off ammonia and continually attracts moisture from the atmosphere. This formation of ammonia takes place more rapidly if the blood meal is mixed with finely powdered aluminium silicates—as, for example, brick dust,—and at the same time freshly burnt wood charcoal in powder be added. The mixture becomes warm, and the warmer the thicker the layer. The addition of wood charcoal prevents the loss of ammonia by condensing the gas in its pores. On this account the addition of charcoal to artificial manures which contain blood meal is much to be recommended, since then no loss of nitrogen occurs. If blood meal

be mixed with dry earth, an increase in temperature and evolution of ammonia can be distinctly observed. If moist earth be used, the blood meal is quickly decomposed, an action which may be still more accelerated by adding alkaline substances, such as wood ashes. Accordingly it is advisable, before using dried blood as a fertiliser, to mix it with wood ashes and powdered charcoal, and to apply it to the ground in wet weather. The decomposition and action of the fertiliser then take place very rapidly. Blood manures are hence very valuable in agriculture, especially since the food they contain can be very quickly conveyed to the plant in soluble form. The author has made experiments on vines and kitchen vegetables, in which very good results have been obtained. For one vine 140 grms. (about five oz.) of dry blood meal, mixed with double the weight of wood ashes and charcoal, are sufficient. This mixture is to some extent mixed with the soil of the vineyard, and is also placed in the pit, where otherwise stable manure would be used. A great advantage of this system of manuring lies in the saving of labour, since one man can readily manure in a day several hundred vines.

It should be remarked that the fertiliser, mixed with an equal weight of sieved wood ashes, may be kept or packed in well-closed vessels, *e.g.* in casks lined with tinfoil, and best in empty petroleum or oil barrels. It should be well stamped down, so that there are no interstices and the atmospheric air cannot act on the blood meal. The mixture must also be kept dry, so that there is no loss of ammonia.

Preparation of Albumin from Ox Blood.—The blood caught fron the slaughtered animals is allowed to coagulate in dishes, when the albuminous liquid separates at the top and can be poured off. During this process the dishes are kept in a cool place, where the separation more readily occurs. The so-called "blood cakes," which are here formed, are brought on a linen filter and gently squeezed, in order to obtain the remaining albumin which they contain. The residue is then cut into lumps and dried in drying-chambers on zinc plates. The blood-albumin is now treated according to the method described for drying the fluid blood, by

bringing it in very thin layers upon zinc plates with turned-up edges, and freeing it from water in a drying-chamber at a temperature which does not exceed 30–35° C. The zinc sheets are previously greased with a little olive oil, in order that the dry albumin may more easily be removed. To free the dried albumin as far as possible from other matters which adhere to it, distilled water is poured over it. After some time this is drawn off, when it contains the readily soluble phosphates. The residue is now mixed with warm distilled water and frequently stirred, when the blood-albumin dissolves. The solution is filtered through flannel, which retains the impurities and the colouring matter of the blood. The filtered concentrated solution is again brought on the zinc sheets and dried in the drying-stove at 30–35° C. The principal application of blood-albumin is in cotton-printing, in order to obtain fast colours.

Edmund Campe, of Brünn, has related certain very noteworthy experiences of the practical manufacture of blood-albumin (Wittstein's *Vierteljahrschr. f. prakt. Pharm.*, 1872). In order to obtain a very pale blood-albumin, it is necessary to use the greatest care in catching the blood from the slaughtered cattle and sheep. Then it is necessary that the position, where the vessels for clearing the serum and the sieves are placed, should be very near to the slaughter-house. When possible, the serum should be drawn off in the slaughter-house itself, or the immediate neighbourhood, and the blood should be brought on to the sieve not longer than thirty to sixty minutes after it was caught. The freshly-curdled blood is cut into cubes of about one-inch edge, brought upon the sieve, and allowed to drain for forty to forty-eight hours. After the lapse of this time, the clear serum is drawn from the clearing vessels, with the precaution that none of the red colouring matter deposited at the bottom is carried away. In order to avoid this, Campe employs clearing vessels with somewhat concave bottoms, and has the orifice for the cork of the exit pipe about one-eighth of an inch above the bottom.

After all the clearing vessels are emptied, the whole of the serum is placed in a tub of soft wood, about three

to four cwt. in capacity. The tub, which is wider at the top than the bottom, has a wooden tap about two to three inches from the bottom. The further treatment of the serum varies, according as "natural" albumin, *i.e.* without shine, or "patent" albumin—which shines—is to be made.

In order to obtain natural albumin, it is only necessary to add a quarter of a pound of (spirits of) turpentine to each cwt. of serum, and stir well for an hour. For this purpose Campe uses a circular board, about one foot in diameter, perforated with holes, and fastened to a stick. The serum is then covered, and left twenty-four to thirty-six hours at rest. The turpentine separates at the surface, mixed with a greasy, greenish white fat. The clarified serum is now drawn off through the wooden tap near the bottom. The first runnings, which are always somewhat turbid, must be rejected; the remainder of the serum is then taken to the drying-stove for evaporation. Campe evaporates in stamped iron trays, 12 inches long, 6 inches wide, and $\frac{3}{4}$ inch deep, which are painted with oil paint, varnished, and stoved. The temperature of the drying-stove, when the serum is poured into the trays, should be about 50° C.; when the trays are full, the temperature may rise rapidly to 52–55° C., which may be maintained for two hours, without opening a ventilator. After this time all the ventilators are opened, and the temperature allowed to sink to 50° C., at which it is maintained until the end. Here and there the ventilators are opened, in order to replace the moist air by dry. In order to promote a rapid renewal of air, Campe employs air-holes in the masonry at the bottom. The exits are naturally at the top, and open above the roof.

In order to produce from the serum the so-called "patent" albumin with a handsome glitter, Campe takes for each cwt. of serum, $6\frac{2}{3}$ drms. of oil of vitriol and $6\frac{1}{4}$ oz. of strong acetic acid (sp. gr. 1·04), mixes the two, and, after standing for one hour, dilutes with about 6 lb. of water, and then pours in a very thin stream into the serum, whilst constantly stirring. Next, $\frac{1}{4}$ lb. of turpentine is added per cwt. of serum, and the mixture well stirred for sixty to ninety minutes. After the serum

has stood twenty-four to thirty-six hours at rest, it is drawn off as before, and ammonia added to weak alkaline reaction before placing in the drying-room, in order to remove every trace of free acid. The plates are rubbed with warm tallow, so that the finished albumin may easily be removed from them.

By this treatment only a portion of the albumin is extracted from the blood for the preparation of the so-called primary albumin. Now comes the manufacture of secondary and tertiary albumin. The second variety is more an accidental manufacture, for which only the serum of those vessels can be used which for any reason has acquired a reddish colour. Campe also took for it the last pale red liquids obtained in drawing of the serum for primary albumin. The treatment for secondary albumin is the same as that given for primary. Tertiary albumin is the last product; it is used in sugar refineries in considerable quantity. The cubes of blood which remain upon the sieves are brought into a vessel with a false bottom about eight to twelve inches up and bored with half-inch holes; water is poured on in sufficient quantity; also, all the residues from the primary albumin are added—*i.e.* the red deposit which formed in the settling vessels. The whole is well worked up with the hands. The liquid which collects below the perforated bottom is made slightly alkaline with a little ammonia, and brought into the drying-stove. This product also glistens, and is the so-called tertiary albumin.

The blood, which remains behind in the double-bottomed vessel, is then passed (according to Campe) between two toothed rollers, working together, so that a uniform paste is obtained, which is dried at 60–75° C. in a stove, built with shelves. The sheet-iron drying-trays used in the stove are 2 feet long, 1 foot wide, and about 1½ inch deep. The use of this product is somewhat extensive. In order to utilise it better as a fertiliser, Campe moulds it, with solid human excrement and ground oak galls, into bricks, which are dried in the air, and finally ground on the manure mill. The product is known as blood-manure: it contains about six per cent. of nitrogen, and is particularly effective for grasses and leguminous plants.

Preparation of Fibrin and Butyric Acid.—The fibrin obtained in the form of threads by whipping ox blood is washed with distilled water until it appears quite white, and the blood corpuscles have been removed. It is then rapidly freed from water on linen cloths, as before described, and dried on hair-sieves at 120–150° C.

If the moist fibrin be treated with pyrolusite and sulphuric acid, butyric acid is formed, which may be used in the *manufacture of butyric esters.*

Butyric acid is first prepared as follows:—80 grms. of moist fibrin, 320 grms. of starch, 20 grms. of tartaric acid, and 5 kilos. of hot water are mixed together; then 1·5 kilo. of sour milk is stirred in, and the mixture left for forty-eight hours in a warm place at 32–36° C., until fermentation is noticed to commence. When the fermentation is well started, 520–550 grms. of finely-powdered chalk are added, and the whole frequently stirred. After fourteen days the formation of calcium butyrate is almost ended; a mass of crystals has formed. The process is finished when no more bubbles of gas are given off. The liquid is then filtered through cloth, and sufficient sodium carbonate added to convert the calcium butyrate into sodium butyrate and calcium carbonate. The latter is filtered off and the liquid evaporated. To the residue sulphuric acid is added, equivalent in quantity to the sodium carbonate; the butyric acid is separated, and sodium sulphate remains in solution. The butyric acid is again neutralised with soda, and the butyrate decomposed by sulphuric acid in a glass retort, when colourless butyric acid distils over.

In order to obtain butyric ester from the butyric acid, two parts of alcohol are mixed with two parts of butyric acid, and one part of strong sulphuric acid added. An oily layer separates upon the liquid; it is washed with water, shaken for some time with magnesia, filtered, and dehydrated by means of calcium chloride. It is finally distilled from a glass retort, when a water-white, very mobile liquid is obtained, of a penetrating odour similar to that of pineapples. The specific gravity of the ester is 0·913; it boils at 113° C.

Butyric ester is principally used in making imitation rum. It is mixed with the proper quantity of rectified spirit, acetic

ester, vanilla essence, sugar ether, and water—in the last of which sugar-candy and caramel are dissolved, in order to give the rum the necessary colour. A very good formula is—for 50 litres of rum, take 500 grms. of finest rum essence, 100 grms. of butyric ester, 100 grms. of acetic ester, 50 grms. of sugar ether, 50 grms. of essence of vanilla, 150 grms. of essence of raisins, 30 litres of high strength spirit, and 30 litres of water. In the water dissolve 1 kilo. of sugar-candy and 250 grms. of caramel; mix the whole well, and allow to settle for several weeks.

Butyric ester is also much used in preparing fruit essences, *e.g.* of raisins, apricots, strawberries, etc., which are again mixed with spirit and other ethers in various proportions.

Fat from Animal Offal.—The oldest method for obtaining fat from animal offal consists in heating in a boiler over an open fire. In employing this method, in order to avoid noxious odour, a simple expedient may be adopted, according to Terne (*Chemiker-Zeitung*, 1879, No. 11). In dealing with pans heated directly by fire, they must be covered by a sheet-iron lid in such a manner that, on the one hand, the workman is not prevented from stirring and skimming off fat, and, on the other, the pan may be tightly closed during the intervals. When the lid is brought into communication with the ashpit of the boiler-fire in a proper manner, the noxious vapours in passing through the fire will be partially decomposed, and partially so diluted with the fire gases that they reach the atmosphere through the chimney unremarked. If the offal is boiled, as is almost universal, by steam in wooden vessels, the noxious odours may be removed in a simple manner. The annexed sketch (Fig. 5) is self-explanatory.

The conditions are different when flesh is boiled by means of high-pressure steam. According to Terne, the simplest and best method for removing the noxious gases and vapours is the following:—The mixture of gases from the closed boiling kettle is first subjected to the full heat of the boiler fire in a superheater, built of fireproof materials, placed in the boiler flue. From this superheater Terne takes the gases, when possible, along both sides of the boiler fire in a hollow space built of fireproof materials, one side of which forms a

wall of the fireplace, and the other side of which is in communication with the atmosphere through short pipes. The superheated gases enter this retort-like space, mix with the atmospheric air, and then the mixture enters the firebox of the boiler. The gases, in this highly heated condition and well mixed with the oxygen of the air, meet the fire and are burnt with absolute certainty. It is to be remarked, that the best apparatus may work without effect if the gases from the kettle are blown off suddenly under great pressure and with the cock full open. In boiling a kettle, the exit pipe must

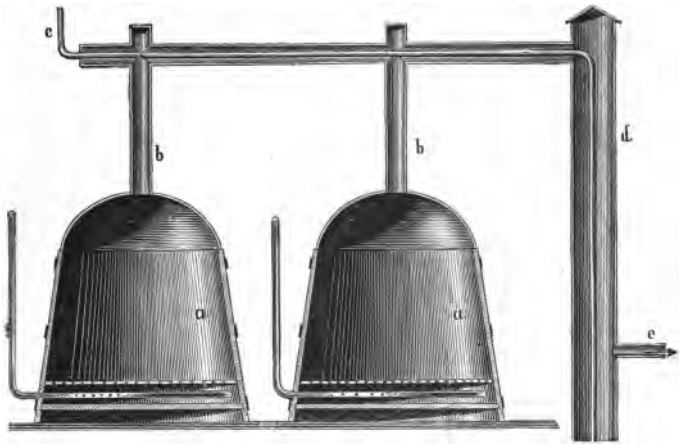

FIG. 5.—*a a*, Pans with perforated false bottoms and perforated pipes for direct steam; *b b*, pipes connecting with a large pipe; *c*, condensing pipe carrying a current of water; *d*, condensing tower with water-pipe; *e*, steam injector to create the draught.

be left open until the boiling-point is reached. When boiling actually begins, the cock is closed and the kettle raised to the pressure at which it is intended to work. The kettle should be allowed to stand at this pressure for about an hour; then, whilst still admitting steam, the blow-off cock is cautiously and slightly opened. With careful management it is possible to keep almost the original pressure. If the gases are removed regularly and slowly, they may be destroyed without fail; but if they suddenly rush with the full pressure of 2–3 atmospheres, with the cock full open, into the com-

bustion apparatus, a great part will naturally escape unburnt into the air.

Extraction of Glue from Animal Waste.—According to Terne's communication on the manufacture of glue (*Dingler's Journ.*, 1876, p. 253), slaughter-house material may be arranged, according to the proportion of glue it contains, as follows:—(1) ox feet, (2) pigs' feet, (3) calves' and sheep's feet, (4) raw bones, (5) ox and pigs' heads.

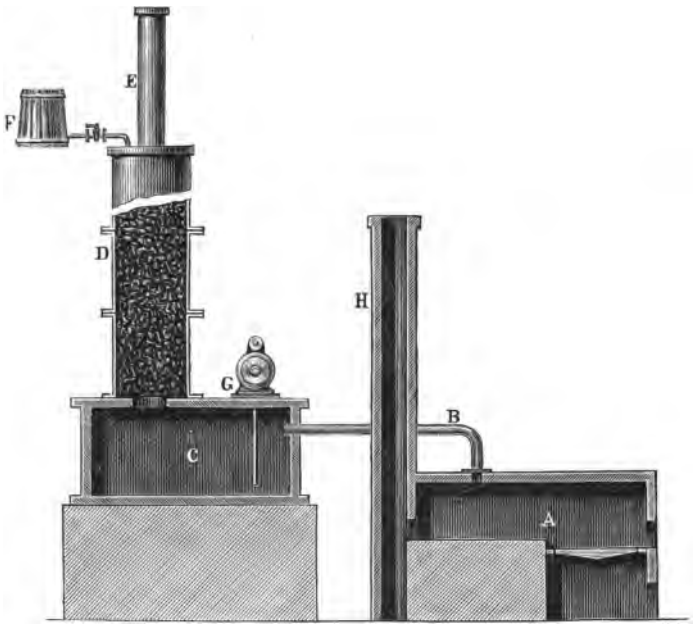

FIG. 6.—*A*, Sulphur burner; *B*, stove exit pipe; *C*, collecting tank; *D*, coke tower, consisting of twelve earthenware pipes, each 760 mm. (30 inches) long; *E*, draught regulator; *F*, water-tank; *G*, steam pump for acid; *H*, chimney to sulphur burner.

The material is to be freed as far as possible from blood, and this will be the easier the smaller the parts. For this reason the larger works have machinery for tearing up the fleshy portions, and breaking up the bones. The best bone-breaker, according to Terne, is that of Baugh & Son, of Philadelphia (*Dingler's Journ.*, 1869, p. 186). When the

material has passed through the breaking machine, it is most advisable to free it from blood and dirt in a suitable washer. After removing the blood, Terne treats the material in suitable wooden vessels, which must be well covered, with a saturated solution of sulphurous acid. The duration of the action varies

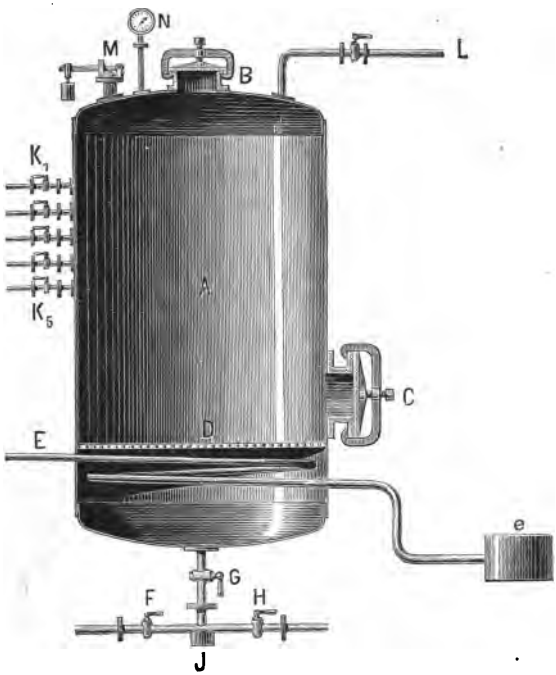

FIG. 7.—*A*, Iron pan, lined with lead in order that the bleaching process may be conducted in it. *B*, feed opening; *C*, outlet; *D*, perforated bottom; *E*, steam coil connected with the steam-trap *e*; *F*, steam pipe; *G*, drain cock, shut during boiling; *H*, cock on drain pipe; *J*, water outlet; K_1–K_3, run-off cocks for fat and oil; *L*, pipe conveying the noxious gases to the boiler fire; *M*, safety valve; *N*, pressure gauge.

greatly according to the nature of the material; experience alone is the only guide by which it is possible to determine the amount of action proportioned to each kind of material and the season of the year. The results are twofold. The material produces a clear, almost colourless liquid, which, after

evaporation in vacuum pans, gives a glue in no way inferior, either in colour or shine, to the best hide glue. The fat of yellowish bones is perceptibly bleached, and has not the unpleasant smell otherwise exhibited by bone fat.

For the production of the sulphurous acid, Terne has constructed a simple apparatus, which has been found practically successful. Coke towers are erected in such positions that the outer wall is warmed by the heat of the boiler-house, and thus the whole is protected from frost. (See Figs. 6 and 7, pp. 42 and 43.)

In consequence of the treatment with sulphurous acid the portions of hide, and especially the sinews, swell, the latter acquiring a silky lustre and becoming transparent as gelatine. The tissues which produce the glue are not only bleached, but also become much less compact. In consequence of this treatment it is possible both to shorten the time of the first boiling of fresh bones, and also to diminish the pressure.

The washed and bleached raw material is now ready for boiling. The chief conditions for the production of good strong liquors are low pressure and the shortest possible duration of boiling. The boiler erected as shown in Fig. 7 satisfies all the conditions required for a regular, gentle boiling, and affords a certainty of obtaining a product almost free from fat. The pipe *F*, for direct steam, is only employed for bringing the water rapidly to the boiling-point; the coil *E* is sufficient to keep it boiling.

The liquor, completely free from fat, must now be filtered, for which purpose Terne employs a bone-black filter filled to a height of about 1·22 metre (4 feet). The bone-black is covered with coarse sacking, and the whole filter is surrounded by a steam jacket. Probably a filter press, such as are made by Wegelin & Hübner, of Halle, would be more suitable. From the filter the liquid is to be taken to the vacuum pans. The consistency of the size to be obtained in the vacuum pans varies greatly according to the purpose for which it is intended, and the temperature of the air; it is impossible to lay down general rules. However, the size must be boiled down to such a consistency that the gelatinised green glues to be obtained must be capable of being cut, or that the glue

to be cast into plates must be readily and quickly removable from the moulds. Strong tinned sheet-iron plates are the most suitable for the moulds, but they have the disadvantage of readily warping. In order to avoid this, glass tables made for the purpose are employed; they give the glue a very fine glitter, but the fragility of the glass plates is very objectionable.

Every manufacturer has to give the greatest attention to the cooling of the glue; when possible it should always be cooled at a temperature of 0–5° C. In order to be able to accomplish this, all the large works in America are provided with ice-houses and cooled working-rooms. For glue which is to be cut, Terne has found moulds of strong galvanised iron, about 3 mm. thick ($\frac{1}{8}$ in.), the most suitable. The moulds are about 254 mm. (10 in.) high, the upper opening is 305 × 356 mm. (12 × 14 in.), and the bottom 290 × 330 mm. (11·5 × 13 in.). In order to economise ice, the glue in these moulds may, with great advantage, be cooled by running water round them. Moulds constructed of bad conductors of heat are absolutely to be rejected, since they tend to make the glue liable to decomposition.

The plates of glue, cast or cut out of the moulds, are then dried on wire netting. For the sake of economy in netting, Terne hangs the plates, after they have attained a certain firmness, vertically from thick iron wires.

CHAPTER VII

MANUFACTURED FUELS

Artificial Fuel.—In the year 1874, Ney, before the Aix-la-Chapelle Society of Engineers, confirmed the statement, originated in Belgium, that a mixture of 80 lb. of soil with 20 lb. of small coal, moistened with a solution in water of 1 lb. of soda or common salt, burnt well. This amount of coal, which by itself would only last fifteen minutes, now burnt for an hour. This slowness of burning is, however, all. It is explained by the formation of glasses from the soda and the earthy constituents of the soil, below the slag thus produced the fire burnt for a long time. Naturally, direct action of the soda on the fuel is not to be imagined. R. Hasenclever, on this occasion, called attention to the high proportion of combustible matters in certain soils. Thus street mud from Stolberg, near Aix, according to his researches, contained not less than 20–21 per cent. of combustible material; whilst dirt from the Römerstrasse, in Aix, only contained 6 per cent. The choice of the soil is therefore of importance. At Hasselt, in Belgium, where the discovery of the above artificial fuel was made, there are many cavities filled with turf-like soil. When such soils are not at hand, the mixture is therefore devoid of importance. The mixture, as was to be anticipated, has been found quite unsuitable for firing boilers.

Manufactured Fuel.—Loiseau (*Zeits. f. d. chem. Grossgew. v. Post*, 1877, 1) prepares a fuel, in lumps of the size of a hen's egg, from 95 per cent. of coal dust, 5 per cent. of clay, and a binding substance made from rye flour and slaked lime. The lumps are then soaked with paraffin residue dissolved in benzine, in order to protect them from the weather. The clay is dried on iron plates over a movable fire and ground,

then mixed with sieved coal dust by means of a special appliance, which measures off the necessary quantity of each constituent, and throws them into a receiver, where they are mixed with the binding medium, which drips over them. In a second large iron tank, the mass is kneaded between iron rollers, with intermoving teeth; it then goes to the hopper of the press, the two rollers (29 inches in diameter) of which each are hollowed out into 870 large and 56 small moulds. When the moulds meet in the rotation of the rollers, the soft mass is pressed in, and falls in egg-shaped lumps upon a moving band of woven wire. The large lumps weigh 3 oz.; the smaller, about ½ oz. They are dried by traversing five times, on a moving woven wire belt, an oven at 250° C., and then fall on to another belt with wire sides which carries them through a bath composed of paraffin residues and benzine. The volatile benzine is then distilled off in the "evaporator," when the briquettes are ready for use. The apparatus turns out 150 tons of briquettes daily. The product is twenty shillings per ton cheaper than ordinary coal.

Briquettes from Lignite.—At the lignite mine at Frielendorf, near Cassel, a new briquette works was erected in 1876 (*Preuss. Zeits. f. Bergwesen*, 1876), the arrangement of which was as follows:—The lignite was drawn up from the bottom of the open working in half-ton waggons in a winding shaft, by means of steam power, to the top, whence it could be transported on a covered tramway, either to the hoppers from which the boilers were fed, or to the hoppers of the rotatory sorting apparatus, and there emptied out. A short screw conveyer carries the lignite which is to be compressed to a sorting cylinder, which passes all the pieces above 7 mm. partly through the large openings in the latter half of the sieve, and partly through the end of the cylinder, whence they fall upon a pair of rollers below. The material, which is here ground below a maximum size of 7 mm., together with the small which has fallen through the finer part of the sieve, passes into a horizonal screw conveyer, which now carries the fine coal to an elevator. The latter raises the coal through a height of 8 feet to a horizontal screw conveyer,

passing over the drying-ovens, which are thus furnished with the necessary quantity of moist ground lignite. After the material has passed through the interior of one of the four sections of the automatic oven, and has left the delivery apparatus, the now dry coal is taken by an elevator, cased with sheet-iron, to the level of the hoppers of the two presses, from which it falls spontaneously through a regulator, and finally leaves the presses in the form of briquettes. These now go in a wooden trough, through which the machine forces them in a compact string, into the store to be stacked, if they are not loaded for transport direct from the trough. The presses do not differ materially in construction from other coal presses which have recently been found satisfactory. Each consists essentially of a thirty horse-power horizontal engine, together with the stamp and the press head, which is rigidly connected with the bed-plate of the engine. The press head—a cast-iron cube—carries the orifice through which the lignite is supplied, and is traversed by a chamber 62 inches long, containing the press feeder, which consists of top, bottom, and two side wedges, between which the stamp moves and effects the compression of the material. A wrought-iron plate resting upon the upper wedge, pressed by a screw at the front, and moving on a strong spindle, serves to close the top of the press chamber and to regulate the pressure or friction in the mould. The lignite already in the press chamber, which has been solidified, provides the resistance for the next following briquette. At both sides of the press block are hollow pieces, through which hot steam is sent to heat the block. The compressed lignite, as previously remarked, from the press enters a wooden trough in front in a continuous stream, and is then pushed outside the building into waggons, or to the store. Without overloading the machine, the stream of briquettes may be from twenty to forty yards long. One great advantage of the Frielendorf apparatus, as opposed to the ordinary trough and plate machine, is that, within the former, there are no moving parts, which in the latter cause so many stoppages and repairs, and also give rise to the formation of much dust and danger of explosion.

Compressed Fuel from Lignite-Coke and Peat.—F. Mathey states that a compressed coal may also be made from lignite-coke and peat; yet these require a somewhat greater addition of saltpetre than charcoal. The latter, obtained from different works, contained 2–4·5 per cent. of potassium nitrate (*Deutsche Industrie-Zeitg.*, 1876, p. 125; *Dingler's Journ.*, 1878, p. 90). The Chemnitz works use as binding medium gum-arabic waste; Knorr, of Weissenfels, uses rye flour; others, dextrine. Of gum-arabic, as much as 4 per cent. is added; of dextrine and rye flour, 4–8 per cent. Instead of rye flour, gluten—a waste product of starch-works—would be better.

Practical Experiences in Briquette-making.—At the Aix District Association of German Engineers, Hilt gave a paper concerning the local development of the briquette manufacture. (Wieck's *Gwb.-Zeitg.*, 1878, No. 39; *Zeits. f. d. chem. Grossgew. v. Post.* III. 1, 1878.) In the first place, he maintained that this industry could only take root where small coal was to be obtained at one-quarter of the price of large coal. Coking is the best method to use with very bituminous small coal. For briquetting the small of non-caking coals, there may be used—(1) mineral binding agents, such as clay, loam, gypsum, cement, and lime; the resulting coke naturally contains much ash and is not hard; (2) carbonaceous binding agents, such as glue, starch, tar, and coal-tar pitch. The briquettes obtained are impervious to weather, may be readily transported or stored without crumbling, and possess a heating effect similar to that of large coal. The first species of briquettes, made with mineral substances, is much used for domestic purposes, but is unsuitable for boiler firing. The second kind, made with organic substances, may also be used as boiler fuel, with a good draught and care in firing, without producing excessive smoke. The somewhat slow combustion of these briquettes may be counteracted by using a thicker layer of fuel, by breaking them up, and by more frequent raking of the fire. In making the briquettes, the coal and binding medium are mixed in the proper proportions in a suitable apparatus, provided with a mechanical stirrer, in which the mass is softened by hot air entering the

apparatus or by heated steam. The briquettes are then moulded, under a high pressure, in closed moulds, or in an apparatus similar to the well-known brick press.

Drying apparatus for briquettes has been made of many forms. Most of these considerably increase the cost of manufacture. The cheaper drying apparatus of A. Wilcke is here described (*Dingler's Journ.*, 221, p. 523). Upon a solid foundation, 3·25 metres (12½ feet) square, in which are two flues for supplying and removing the heating gases, are built two walls containing flues, 1 metre (39 inches) thick and 1 metre apart. Between these two walls floors are built in, consisting each of two iron plates, which floors are closed at the short ends by plates, so that the fire gases introduced by the flues into the closed boxes must heat the plates. Thus, when the flues are connected to a fire, they bring the gases to the second floor, which they pass through, then through the third, and so on, until they are drawn off to the chimney and reach the atmosphere. If eight such floors, each 3 metres long, are placed one above the other, the fire gases will have an opportunity of giving up their heat to the 24 metres length of iron plates which they touch, to such an extent as is allowable in view of the necessity of producing a sufficient draught in the chimney. The gable-ends of the iron floors are protected from cooling in a suitable manner. The separate floors are so arranged as to project in turn, so that the projecting part catches the coal falling down from the floor above. Similarly, in order to shut off the open space between the separate floors from the outside, movable flaps are provided. The upper plate of each floor is surrounded on three sides by a ridge; on the open side the coal is brought down to the next floor. Thus, as was said above, the coal goes from floor 1 to floor 2; the narrow edge of floor 2, protected by the ridge, projects 250 mm. (10 inches) beyond floor 1. If now some arrangement be provided by which the coal, which reaches floor 1 from a hopper, is moved over the plate of floor 1 to that of floor 2, and so on, and if this motion be so regulated that the coal is heated to about 60° C. when it arrives at the delivery-shoot, coal quite suitable for briquetting will be obtained.

The movement of the coal over the plates is accomplished by means of a lattice provided with scrapers, and moved by mechanical power. Two ⊥-shaped rails are connected by two crossbands and two transverse rails; this grating is provided on the flat under-surface with flat scrapers 100 mm. (4 inches) apart, and is coupled to cranks on the shafting. When the shaft rotates, the grating is moved backwards and forwards over the plates. This motion to and fro would, however, not move the coal lying on the plates, which can only be done if the grating, in moving back, is raised over the coal without touching it. Projecting pieces are arranged on the shaft, which act so as to raise the grating in the reverse movement. This rise is naturally at first gradual, so that the scrapers level the ridges into which the coal was brought in the forward movement. Thus the coal is stirred, which is a necessary condition for the uniform drying of coal intended for briquettes. The grating is supported by a foot running on a small wheel, so that it may keep at the greatest height until the end of the reverse stroke; when the reverse stroke is finished the grating falls, and then again moves the coal forward.

The rails run upon rollers, in order to prevent the scrapers from grinding on the plates during the forward movement, and stays are used in the floors to prevent the plates from buckling. The grating also runs upon three wheels in the middle.

If now coal is continuously brought upon floor 1 from the hopper, when the grating is put in motion the coal is regularly moved forward. As soon as the grating commences the retrograde motion a valve closes the hopper, and feeding ceases until this motion is finished. If this were not done, too much coal would be piled up in the hopper, and some would be thrown over the edge. The coal is gradually carried over floor 1, then falls to floor 2, and so on, until it finally reaches the press, whither it is carried by elevators or conveyers. During the passage of the coal it is uninterruptedly exposed to the heat radiated by the plates, which is prevented from escaping too rapidly by the flaps before mentioned. The fresh coal is subjected to the greatest heat, since the fire gases are conducted into the apparatus from above.

Clayton has constructed a machine for compressing small coal into briquettes (*Dingler's Journ.*, 203, p. 271). A horizontal plate carrying moulds of the required size is placed between two cast-iron columns. Upon the plates moves the filling-box, to which the material, prepared in the proper manner, is conveyed from the mixing-drum. The filling box obtains its movement to and fro by means of a bent lever from the cross-head. The briquettes are compressed from two opposite sides. The lower pistons are driven by cogs on the wrought-iron shaft supported by the framework. The upper pistons of the press are actuated from the shaft by cranks and strong connecting rods, suspended from the crosshead which moves in the framework. In order to avoid excessive pressure on the mechanism, the upper pistons are provided with strong springs.

When the moulds are filled with coal, the upper and lower pistons at once move and compress it to a solid brick. The upper pistons then rise, and are followed by the lower pistons, the finished briquette being thus raised out of the mould to the level of the plate. The filling-box in its return with fresh material removes the briquette. The reversal of the lower pistons, which also effects the lubrication of the moulds in a well-known manner, is brought about by cog-wheels on the shaft.

A two horse-power steam-engine is sufficient to drive a press which can produce per day about 10,000 briquettes, each weighing 5 lb. The space required is very small; a ground space 5 feet long by 4 feet wide, with a clear height of 6 feet, is sufficient.

Finally, E. F. Loiseau has constructed a machine for converting anthracite dust into an artificial fuel (*Journ. Franklin Institute*, 1873, p. 266; *Dingler's Journ.*, 210, p. 437). The coal dust is moistened on a platform and then scraped into the hopper of a stationary cylinder, in which a shaft rotates. On the shaft are six radial plates which divide the interior of the cylinder into six equal spaces. The coal dust fills these spaces, and the rotation of the plates drives it into an opening beneath, through which it leaves. A smaller hopper, placed close against the first, receives clay

previously dried and ground. This passes through a smaller cylinder, also provided with rotating partitions, and is then emptied into the same channel as the coal dust, with which it mixes.

The space between the partitions of the clay cylinder is calculated regularly to take and deliver 5 parts of clay, whilst the larger cylinder delivers 95 parts. The mixture of coal and clay is sprinkled with milk of lime whilst it is falling below a chain elevator, which now raises the moist mixture to the hopper of a conveyer. The Archimedean screw rotating in the latter drives the material into a mixing apparatus, where it is rapidly transformed to a plastic mass by means of seven vertical shafts, to each of which four toothed arms are screwed. These arms cross one another in all directions, and intimately mix the coal and clay together. Through suitable openings at the bottom of the mixer, the plastic mass falls along a shoot into the kneading machine, in which, by a series of knives attached to the central shaft and a propeller, it is forced through an opening at the bottom between two rollers, in the periphery of which are series of oval moulds. These rollers, moving in opposite directions, take the material presented to them in the form of a coherent band, and mould it into oval lumps. An endless wire band carries the lumps away and delivers them into a hopper placed above the drying-flue.

The drying-flue is heated from a fireplace at each end. It contains five endless wire belts, one above the other, which move in opposite directions over rollers placed at the ends of the stove, and which have an arrangement for preventing the lumps from falling down. The compressed lumps of coal fall upon the topmost wire belt, are carried by it through the whole length of the stove, and then slide along a shoot which carries them on to the second endless belt beneath. This belt carries them in the opposite direction through the whole length of the flue to a second shoot, from which they pass down to the third belt, upon which they are carried forward, and so on. The last wire belt carries the coal out of the stove and empties it into the buckets of an elevator, by which it is raised and then delivered to another endless band, which

passes through an open tank, fed continuously from a neighbouring larger tank, by which the smaller tank is kept filled with the waterproofing composition. The endless band is provided with small partitions, which prevent the oval lumps from suddenly falling down into the composition. The continuous immersion of the coal in the waterproofing mixture is brought about by small balls at each side of the belt, which run in narrow grooves on the walls of the tank. On leaving the tank the excess of liquid drips from the lumps of coal through the wire belt into a gutter beneath, from which it flows away by a pipe to a convenient tank.

The waterproofing composition employed by Loiseau is a solution of colophony or some other resin in benzine. In order rapidly to evaporate this benzine, the lumps of coal coming from the bath are emptied into the hopper of a stove of smaller dimensions than the drying-flue, in which only three endless belts are placed one above another. In this flue a strong current of air, driven by a fan, rapidly evaporates the benzine, whilst the coal goes from one belt to another. From the last belt it falls through a shoot into the coal truck outside. The end of the shoot may be raised so that a filled truck may be taken away and an empty one brought in its place.

During the passage of the material through the flue the hot air acts on all the lumps and rapidly dries them. The whole process of manufacture, from the passage of the coal dust into the first hopper, proceeds automatically: coal dust and clay are mixed in the proper proportions with milk of lime, this mixture is kneaded, compressed into oval lumps, which are dried and waterproofed, the benzine is evaporated, and the finished product delivered to the trucks, all mechanically. During the whole process the coal is continuously in motion.

CHAPTER VIII

WASTE PAPER AND BOOKBINDERS' WASTE

Utilisation of Waste Paper.—The waste paper (*Papierzeitung*, 1886; *Neueste Erfindungen und Erfahrungen*, 1886) is thrown into a conical drum, about 10 feet long and 3 feet in medium diameter, covered by wire netting of three-quarter inch mesh; in passing through this drum the greater portion of the adherent dust is lost. Bones, pieces of wood, and other large hard objects, which announce their presence by striking the drum, can be removed by the workman who takes away the waste paper which has gone through the drum. On account of the dust, this sieve works in a casing lined with boards; it may be fed through the open narrower end or through a hopper; thirteen to twenty revolutions per minute are sufficient. The paper is next ground under edge-runners, and is then passed through a second sieving drum, somewhat smaller and covered with a No. 5 or 6 sieve. This drum is closed at the wider end (the exit) by a wooden bottom, which has two openings closed by slides. By this means the ground paper waste may be kept in the drum until all the particles of paper have passed through the sieve.

The residue, containing rags, thread, etc., which may be sorted and worked up, comes out when the slides are opened. This sieve, like the first, works in a wooden casing, one side of which is open, so that the sieved paper particles may be removed. It is driven at the side by bevel cog-wheels, so that it may be easily emptied; the speed is forty to fifty revolutions per minute. There is no loss of fibres in this method of purification.

Utilisation of Bookbinders' Refuse.—The waste produced in bookbinding may well be used to prepare papier-maché for bas-reliefs, dishes, urns, picture frames, clock cases,

etc. For this purpose all kinds of paper cuttings and waste cardboard may be employed. A second constituent is well-sieved wood ashes, especially those of hard wood; finally a third constituent is flour paste. The papier-maché is prepared from these substances in the following manner:—The paper clippings and other bookbinders' waste are torn up small, thrown into a vessel filled with water, and left to dissolve. Frequent stirring accelerates solution. The dissolved mass of paper is finally removed from the vessel, the water is lightly squeezed out, and the mass then comes into a mortar, where it is well pounded. Next it is taken out, laid on a strong linen cloth, and the water squeezed out as completely as possible. The balls of material obtained are then dried in the sun, near a fire, or in a stove. When dry, the balls are rubbed on a grater, when the particles of paper become similar in feel to cotton. They are then mixed on a board by means of a wooden spatula with ordinary flour paste and spread out with the rolling-pin, just as dough is worked.

This mixture with flour paste, which must amount to one-third of the whole quantity of material to be made, is formed into a ring on a board or table. Two parts of fine sieved wood ashes (best from hard wood) are placed in the middle, water is gradually added and mixed in until the ashes are thoroughly moistened. Finally the paper pulp is worked up with the wet ashes.

The mixture of the three constituents is now brought into the mortar and well pounded; it then forms papier-maché, and can at once be used. If the mass is to be kept moist for a length of time, it is filled into glazed earthenware vessels, which are placed in pairs, one upon the other, and protected from sun and heat.

From this papier-maché bas-reliefs can easily be made, the mass being used in place of the wax which otherwise is employed in embossing. For this purpose a portion is taken out of the earthenware vessel, in which it is preserved, spread out flat to the requisite dimensions for the bas-relief, one side of the sheet so obtained is covered with flour paste, and the mass then pressed upon a smooth surface—slate,

polished wood, metal, or smooth pasteboard. Then a linen cloth folded in two is laid on the mass and superfluous moisture removed by repeated applications of the cloth.

After this treatment the design of the low or high relief can very easily be engraved on the mass by the embossing style; hollows can be dug out, and projections formed by adding fresh material where required.

This mixture has certain advantages over embossing wax. In the first place the style works much more easily in the soft paste, since the mass may be depressed, moved to all sides, raised, and moulded as desired. Should some spot begin to dry somewhat too soon, it is brushed over with a brush dipped in water, when the mass may be worked as before.

If the work is quite dry before it is finished, it may be moistened over a portion, or the whole, of the surface and further worked with the style.

After the finished relief is quite dry it is pasted over with thin flour paste by means of a camel's-hair brush, and then again allowed to dry, when the whole object is polished with a bone polishing style. The object is now in a condition in which impressions of it in wax may be taken. It is then coated with thin size, again dried and polished as before. This last treatment of the surface permits of the application of oils or water paints, and of gold leaf simply by breathing on the surface or by using oil varnish. After painting or gilding the object it is usual to give it several coats of spirit varnish. After the latter is dry, neither heat nor cold, moisture, dust, or fly-spots can harm the work, for the varnished surface entirely protects the surface beneath from injury. Dust is removed with a soft brush and fly-spots by a damp cloth.

Utilisation of used Paper Cases.—By paper cases in this instance are understood the small conical hollow cylinders of hard paper, similar in shape to cigar holders, which are used in the textile industries as yarn spools by weavers and stocking-knitters, and which are made in hundreds of sizes for various purposes and in different forms.

E. Höfel, of Gruna in Saxony, has patented a process for

utilising the large quantities of these paper cases in preparing articles of utility and ornament—umbrella and walking-sticks, fire-screens, baskets, card-tables, toy furniture, picture frames, etc. The cylindrical, or rather conical, form of the cases makes their utilisation easy. Just as the spindles on the spinning-frame must be exactly alike, so also the paper cases originally intended and solely used for the spindles are also exactly similar. One fits over another; the pointed cases may be pushed easily one into the other, and so form a rod already firm and of any required length.

After a number of the cases have been pushed one into the other, and a rod of the desired length obtained, a wooden stick or a wire of sufficient strength is drawn through the tube, in order to give it more solidity and afford a means of attaching the ferrule and handle. The ferrule and handle are screwed on the core at the bottom and top, and confine the bottom and top cases. The stick may then be painted and varnished. A pen-case is simply made from a large paper case, which is altered to fit its purpose by fitting a stamped metal bottom and a similar lid, and then ornamented by paint and varnish. In making containing vessels out of the cases, the so-called "ring-ousel" cases are laid near together and united at top and bottom by a ring of wire. They can then be ornamented in any required colour and a decorated top and bottom, preferably of sheet-iron, applied.

CHAPTER IX

IRON SLAGS

Slag Wool.—In 1875 a loose substance similar to the finest natural cotton was brought into the market by several ironworks—by K. Marien, of Zwickau, and G. Marien, of Osnabruck. The substance differed from cotton in having straight (not curled) fibres, in being shining and somewhat less soft to the touch. The new product was produced by sending steam into a jet of fluid slag.

Slag wool has hitherto been produced only from blast-furnace slag. According to Schliephake (*Polyt. Journ.*), it is best made by blowing with a current of steam, whilst the slag is conveniently drawn off from the furnace through the Lürmaun slag-mould. It is a considerable drawback to the operation that the many fine fibres floating in the air seriously injure the workmen.

Slag wool is utilised in accordance with its properties, of which the most important is its very low conductivity for heat. In covering steam-pipes, the slag wool is laid on the pipes in pieces of about 1000 sq. cm. (160 sq. in.) with a firm pressure from the hand, without beating. A thickness of 8 cm. (3–4 in.) suffices for most pipes. The slag wool, compressed to this extent, is bound round with twine or wire, so that it adheres to the protected pipe. After one or two yards of a steam-pipe have been covered in this manner, the whole is sewn up in coarse sacking, which may then be coated with tar, to prevent the penetration of water; wet slag wool is a better conductor than dry, which is also the case with other bad conductors. For one square metre of surface protected in this manner, about four kilos. of slag wool are required (9 lb. per sq. yd.). A steam-pipe or cylinder so covered is hardly perceptibly warm on the outer surface.

From another practical source it has been stated that the covering of sacking is little used, since it is soon destroyed by the action of light and heat, even when tarred.

It is recommended, in covering pipes with slag wool, to make use of a cylinder of sheet-iron, the diameter of which is 150 mm. (6 in.) more than the external diameter of the pipe. The sheet-iron cylinder is 300–400 mm. long (1–1¼ ft.), and is divided lengthways into two halves, which are bound together by splints, or similar means. Each half has a handle in the middle. This sheet-iron cylinder is placed round the pipe in such a position that one end is closed by a flange; the slag wool is then forced into the space between pipe and cylinder, which is about 75 mm. (3 in.) wide. When the interspace is filled, during which operation too hard stamping is avoided, the sheet-iron cylinder is moved forward with a gentle to-and-fro rotatory motion by means of the handles, and the covering of slag wool wrapped with wire as it becomes exposed. The forward movement of the cylinder and the wrapping with wire must keep pace. The whole surface of the slag wool is then moistened with tar, and then covered with a coating of cement about 5 mm. (0·2 in.) thick. About 10–12 kilos. of slag wool are required to cover a surface of one square metre to a thickness of 75 mm.

The fireproof nature of slag wool, combined with its low conductivity for heat, makes it a very suitable substance for the lining of fireproof safes. Wolpert, of Kaiserslautern, has first drawn attention to its use as a filling material for floors and wainscotting in buildings. "Slag wool," he says, "generally, at present, contains calcium sulphide, which is decomposed into calcium carbonate and sulphuretted hydrogen by the action of the carbonic acid in the air and the water which obtains admittance in washing the wainscot. Sulphuretted hydrogen is known (when breathed in quantity) to be a poisonous gas; accordingly slag wool for such purposes must be examined for calcium sulphide. If vinegar be poured on slag wool moistened with water, the well-known smell of sulphuretted hydrogen—that of rotten eggs—is perceived, if the wool really contains calcium sulphide.

But these opinions against the use of slag wool as a filling for floors, etc., go too far, according to Wolpert himself (*Polyt. Zeits.*); the normal quantities of water and carbonic acid in the air can barely produce a perceptible evolution of sulphuretted hydrogen. Apart from its very bad conducting powers, the layer of slag wool has also great permanence, can withstand a considerable temperature, and is not liable to decay through the action of moisture or gases.

Slag Stones. Slag Bricks.—The production of slag stones dates from 1860. At Osnabruck, at that period, the fluid slags were allowed to fall, like molten lead in the shot-towers, from a height of about eight feet into water, where it formed large bean-shaped lumps, which were used, instead of road-metal, as ballast on railways. The same thing had been done for a long time in England, where the slag was broken with a Blake's stone-crusher, and used for macadam. In particular, the slags obtained in the Bessemer process, when the pig-iron was made from spathic ironstone, are said to be suitable for producing artificial paving-stones, in consequence of the lime they contain.

More recently the best method for this utilisation of blast-furnace slags has been attained by granulating. C. Paschen has published a communication on this subject in the *Notizblatte des Vereines für Fabrikation von Ziegeln.*

The product, the so-called slag gravel, can not only be used with the best success as the embedding material for railway sleepers, but it produces, when mixed with lime in the proper manner, the material for an excellent slag building stone and slag mortar.

The preparation of slag gravel by granulation simply consists in allowing the fluid slag to flow into water, where, by the sudden cooling, purified slags harden to a pumice-like gravel and less purified slags to coarse grains.

Slag stone is in no way inferior to good brick, which it surpasses in fineness of colour and porosity. At first friable, it rapidly hardens in the air. This hardening continues for a long time, even after the stone is used, and since the mortar is also mixed with slag gravel, the stones are completely

bound, so that after several years a wall consists no longer of separate stones, but of one uniform mass.

At the ironworks of Buderus at Lollar, near Giessen, building stone is made from blast-furnace slag in the following manner (Report by the engineer Alberte, *Gewerbebl. f.* Hessen, 1893):—A portion of the hot fluid slag from the blast furnaces is cast in large round iron vessels standing on trucks. These go to the portion of the works where the compressed stone is made and after cooling are emptied. The blocks of slag gradually fall on contact with air to a fine bluish grey meal—slag meal. The greater part of the hot fluid slag, however, runs into a channel, in which is cold water in rapid motion. The slag is thus granulated; the sand produced is mechanically scooped out, brought into the waggons of a wire-rope tramway, which take it to the working place, where they are automatically emptied. Foundry slags have been found to be the best, for they produce the lightest stone. In producing the compressed stone, the slag sand is mixed with the above-mentioned slag meal and milk of lime in the following manner:—A mixture of 2 parts of sand and 1 part of meal is moistened with milk of lime, the moist mass is shovelled into presses and moulded into stones. The automatic steam lever presses of Bernhardi's Son, G. F. Draenert of Eilenburg, have been found to work especially well.

The stones are made in two sizes,—the small size 25 by 12 by 6·5 cm. (10 by 5 by 2·6 in.), and the large size 25 by 12 by 10 cm. (10 by 5 by 4 in.). They have a bluish-grey colour, and are fairly porous. The two horizontal faces are somewhat hollowed out, in order to diminish the weight of the stone and to admit a larger quantity of mortar with apparently narrow joints. The weight of one stone is, air-dried, about 3·3 kilos. (about 7·4 lb.). They are destroyed by a load of 70 kilos. per sq. cm. (1000 lb. per sq. in.). They take up a large quantity of water, and have not a great resistance to the action of fire—a red heat. The stones are not burnt, but only dried in the air.

The slag sand by itself is a very useful addition to mortar. A useful rough plaster is obtained from a mixture of 3 parts of slag sand, 2 parts of slag meal, and 2 parts

of clay, whilst for fine plaster a mixture of 1 part of slag meal, 2 parts of white lime, 4 parts of river sand, quarried sand or slag sand, is recommended. The sand for this purpose must be finely sieved.

F. Kirrmeier, of Speyer-on-Rhine, prepares moulded stones from blast-furnace slag (German patent, 1895). The slag is cast in long sticks notched at suitable intervals, so that when the separate blocks are broken off, stones with granular crystalline ends may be obtained. The moulds used in this process are made up of separate walls with an open side, through which the slag can enter. On the under surface of the cover, on the bottom and on the sides, are triangular ribs, which facilitate the separation of the long blocks into separate stones after cooling.

The slag brick owes its present position to improvements in brick presses and to the use of dry disintegrated lime, which is added to the slag sand (containing as much as 40 per cent. of water) as required. Although the granulated slags, on account of the soluble silicic acid they contain, would harden if pressed or stamped alone, yet they do so more rapidly if lime be added. The stone hardens through the formation of calcium carbonate, as does ordinary mortar, and especially through the formation of solid compounds between the soluble silica, the slag, and the added lime.

Slag bricks are made in the following manner:—The hot fluid slag is conducted into a suitable cast-iron channel, through which a sufficient stream of cold water is flowing. The slag is chilled and falls partially into sharp sand, partially into very brittle lumps, the latter readily broken by a slight pressure. The slag sand passes into catch boxes, from which it is taken by baskets with perforated sides and conveyed to the brick-moulding works. It is mixed with the milk of lime as follows:—The slag sand is shovelled into the stirring machine, whilst the proper quantity of milk of lime at 8–10° B. is run in. The mixture is then moulded in the brick press. The slag sand, mixed with lime, passes into a holder from which the necessary quantity is taken by two pistons into the press. The bricks are ejected from the press, taken by labourers,

and placed in a tram waggon. They dry in eight days. Dark slags have not been found as suitable as pale slags for the production of bricks.

The slag sand is removed from the water basin after granulation by means of a strong scoop-wheel, which is constructed as follows:—The scoops are fastened by screws between two large cast-iron rings, which are hung on two friction rollers with a common axis. The latter is driven by gearing from a small steam-engine built on to one of the four cast-iron pillars. The external periphery of the friction rollers works on the inner periphery of the rings and turns the scoop-wheel, which is prevented from oscillating by two guide rollers at the side. The internal diameter of the rings is such that a line of rails goes through the scoop-wheel, so that a waggon may be pushed in from one side and out at the other when full. The two cast-iron rings, together with the twenty-four scoops, weigh about 90 cwt. The weight of slag to be lifted on at once is about 6 cwt. at most. The apparatus makes a revolution in about five minutes with the engine running at eighty revolutions per minute, so that the arrangement can load 72 cwt. of slag per hour. The rings, and also the columns which support them, are cast at the furnace, and need no further working. The feet of the columns are so wide as to offer a sufficient surface for the foundation, and are therefore built on the masonry of the water basin. The spindles of the steel driving shaft, upon which hangs the whole weight of the scoop, run in ball bearings, which are carried by a wedge in a fork and are adjustable. The fork hangs between the U-pieces which rest on the columns and form the framework of the machine.

Blast-Furnace Slags as Paving-Stone.—It has been found that blast-furnace slags make a good paving-stone. The Altona paving-stone, made from iron slags, consists of powdered slag, mixed with clay or loam as a binding agent, compressed and burnt to vitrification.

The paving-stones are laid in a bed of sand only 8 cm. deep (3·2 inches), stamped down with an ordinary wooden rammer, and the joints then filled with sand and water. The

method of laying may thus be called easy and rapid. These stones do not wear hollow like sandstone, or smooth like granite slabs; they can readily be taken up and laid aside without suffering any injury, which is important when excavations are made for gas and water pipes.

The price of these slag stones varies, according to the state of the market, between 225 and 280 marks per 1000. To this is to be added the cost of discharging, loading, and transporting to the destination, which may be taken at 15 marks per 1000.

Glass from Blast-Furnace Slags.—According to a patent of Basley Britton, at the ironworks of Chesland & Fisher at Wellingborough, the fluid blast-furnace slags direct from the furnace are worked up into glass, they are conducted into a tank holding three-quarters of a ton, and there mixed with other suitable materials. The glass is said to be quite transparent, and extremely soft or plastic; it resists acids, is readily cut by the diamond, and is quite suitable for rough sheets for roofs, skylights, etc. It is cheaply obtained, since the heat contained in the slags is also utilised in the process.

Salts from Blast-Furnace Slags.—According to a French patent (Post, *Zeits. f. d. chem. Grossgew.*), aluminium sulphate, gelatinous silica, and calcium chloride can be obtained from blast-furnace slag. The slag, in fine powder, is treated with hydrochloric acid in an acid-proof apparatus, the acid vapours evolved being condensed in a sandstone receiver filled with water. The acid solution, containing dissolved silica, aluminium and calcium chlorides, is drawn off, diluted with water, the silica allowed to settle, the liquid decanted off, the alumina carefully precipitated by pure calcium carbonate, the precipitate washed, dried, and converted into aluminium sulphate by boiling sulphuric acid. The gelatinous precipitate of silica is purified by washing, and the calcium chloride solution, remaining after precipitation of the alumina, is evaporated.

CHAPTER X

EXCREMENT

Utilisation.—Although there is no want of proposals for the rational utilisation of excrement, yet hardly any process has succeeded in surviving. Thus Leube's process—the application of sulphuric acid—failed, and Petri's manufacture of fæcal stone could obtain no practical hold. The chief aim, after the suitable removal of the fæces, is to utilise them as manure. The preparation of an illuminating gas from excrement is described in the chapter on "Illuminating Gas from Waste." In regard to other proposed methods, with the exception of the manurial, we give the opinion of Fischer (*Dingler's polyt. Journ.*, 213, p. 259):—"As a matter of fact, the application of human excrement as a fuel—Petri's fæcal stone—is the worst imaginable, since its most valuable constituents, nitrogen compounds, are lost. For large towns there is no other course than a well-arranged system of sewers and flushing."

Scott has patented the following process for treating sewage:—The sewage, collected in a tank, is mixed with excess of caustic lime, the clear supernatant liquid is drawn off from the precipitate, and, in a second tank, an iron or aluminium salt added, which salt is converted into the hydrate by the lime present in the clarified water. The precipitate in the first tank, which contains the organic and inorganic matters suspended in the sewage, and to some extent also substances previously in solution, may be converted into cement or filter coke. The precipitate in the second tank, after calcination, gives metallic oxides which may be utilised. The water from the second tank is sufficiently pure to be passed into a large stream, and may, if it has previously been filtered through the coke obtained from the first tank, even be run without danger into a small stream.

CHAPTER XI

COLOURING MATTERS FROM WASTE

Utilisation of the Waste from Dye-wood Extracts.—Croissant and Bretonière, of Lavalle, gave a detailed account of their work in 1875 (*Bull. Soc. Ind. Mulhouse; Dingler's Journ.*, 215, p. 363). In the first place, this concerns the solid dye-wood extracts, each of which contains its own peculiar tannin. Now gallic acid, a product of the decomposition of tannic acid, on heating to about 250° C., gives metagallic acid. The chemists named have therefore treated logwood extract by the same reaction, when they obtained an evolution of carbon dioxide and a black voluminous substance, insoluble in water, but readily soluble in alkalis, and precipitated from these solutions by acids in the form of brown flocks. With different metallic salt solutions it gave differently coloured precipitates. This decomposition of logwood extract, also accompanied by the evolution of carbonic acid gas, takes place at 200° C. in the presence of caustic alkalis. In this case there is obtained the soluble alkali salt of an acid analogous to metagallic acid, which is precipitated from its solutions on the addition of other acids or of metallic salts. The important point is that the alkaline solution of this compound has a very strong direct dyeing power for vegetable fibres. If the natural colouring matters be left, and the same process applied to other organic substances, there is known to result by the action of alkalis a salt of oxalic acid, *e.g.* from sawdust. The process, however, takes quite a different course if sulphur be at the same time introduced into the compounds. Either the sulphur enters into direct combination with the substance without the elimination of any element, as in the case of the aloe, or, as more frequently happens, the sulphur unites with a portion of the hydrogen of

the organic substance to form sulphuretted hydrogen, which is volatilised, the organic substance thus losing part of its hydrogen. In both cases, however, and from almost all organic materials, there are produced in this manner new substances, which are substantive dye-stuffs for animal and vegetable fibres, and produce intense and fast shades without the assistance of a mordant.

The discoverers have heated a series of the most diverse organic substances in closed vessels with sodium mono- and poly-sulphide, and in every case find confirmed the correctness and general applicability of their discovery.

The colouring matters are readily and certainly produced in the form of a swollen voluminous mass, more or less dark in colour according as the temperature of preparation was higher or lower, between the limits of 200–300° C., and according to the longer or shorter duration of the heating. The solubility of the product also increases with this temperature and duration of heating, as also the fastness of the effects produced on fibres, especially in regard to the action of light. All the products are very hygroscopic, and must be kept in well-closed sheet-iron boxes, especially to guard against oxidation by the air, by which the colouring matter is converted into an insoluble substance. Without this precaution they become quite useless in four to five months. In a freshly prepared dye-bath the colouring matter has such affinity for the vegetable and animal fibres that, when the dyeing is sufficiently protracted, the whole may be withdrawn from the bath, and a colourless liquid left. Of particular importance to the productiveness of this colouring matter is the nature of the water employed. In waters containing lime it dissolves only incompletely; if only such water can be obtained, it must be purified before use by boiling with soda. The colouring matter is also separated from its solution by acids, but the precipitate readily redissolves in dilute alkaline liquids. This behaviour affords a means of purifying the colouring matter, and obtaining it in the form of a dry unalterable powder, soluble in alkaline liquids. Other precipitants are alum and metallic salts; the precipitates vary in colour according to the metal. The most important pre-

cipitant for dyeing purposes is, however, potassium bichromate, which is principally important because of the oxidising action of the chromic acid, and because the precipitates which it produces, with slight exceptions, are quite unacted upon by most solvents, even by boiling caustic liquors, so that this salt is a most valuable means for fixing the colour upon the yarn or fabric.

CHAPTER XII

DYERS' WASTE WATERS

Recovery of Arsenates and Phosphates.—For the recovery of the arsenates and phosphates from the solutions used for fixing the mordants—the so-called "dunging"—the following process has been patented by Higgins and Stenhouse:—The waste liquors are mixed with an iron or manganese salt, the mixture made alkaline by milk of lime, and allowed to settle. The precipitate, which contains the arsenic and phosphorus, after removal of the clear liquid, is brought on to cloth strainers. A portion is then examined for the percentage of bases it contains, and an equivalent quantity of sodium monosulphide added to the whole bulk. The mixture is then boiled with water in a steam-jacketed pan for two hours. The resulting clear solution contains sodium arsenite, arsenate, and phosphate; if a little sodium sulphide should be present, it is oxidised by means of sodium hypochlorite. The solution is now again available for "dunging"; if it should be too alkaline, it is neutralised by a mineral acid.

Recovery of Dyes from the Waste Liquors.—This patented discovery is due to Remmers, of Glasgow. It accomplishes the recovery of alizarine and purpurine from the waste liquors obtained in dyeing Turkey reds and other shades, for which madder or artificial alizarine is employed. For this purpose the waste liquors are run into a large tank, into which hydrochloric or sulphuric acid is run in quantity sufficient to precipitate the whole of the colouring matter out of the solution. The reaction is accelerated by stirring. After settling, the clear liquid is drawn off from the precipitate, which is boiled for some minutes, when necessary, with a little acid, until the liquid acquires a yellow colour.

After cooling, the precipitate is separated from the liquid by means of a filter press, and washed until neutral, when it may again be used in any dyeing process.

Recovery of Tin from the Waste Waters of Dyeworks.—Mogret has given an account of the recovery of tin from the waste liquors of dyeworks (*Moniteur de la Teinture*, 1889; *Färber-Zeit.*, 1889). To a greater extent than ever before, tin preparations, in the form of stannic or stannous oxide, play an important part in dyeing, both as mordants and for weighting silk. Hardly half the quantity of tin used is really utilised, the other half is lost in the used mordanting or dyeing baths or in the wash waters of yarns and fabrics. Since this metal is expensive, the wash waters and used liquors represent a considerable sum. In order to regain a portion of the valuable constituents, which run into the drains and contaminate the rivers, the dye-baths and wash waters are collected in tanks. In these the tin is precipitated by the addition of granulated zinc, or, better, of zinc dust—known under the name of "preparation." This tin is mixed with stannic oxide, filtered through woollen cloth, the precipitate washed, dried, and melted at a white heat, with the addition of borax and a little granulated zinc. The stannic oxide is reduced by the zinc; excess of zinc is volatilised at the high temperature employed. In this manner pure metallic tin is obtained, which collects to a lump in the crucible, and is of considerably higher value than the zinc used in its recovery. This method is said to be employed in several large works in France.

CHAPTER XIII

FAT FROM WASTE

Recovery of Neat's-foot Oil.—The fresh feet of oxen, calves, and pigs are boiled in a pan with water; the boiling is continued for fifteen minutes, after which the heating is moderated, so that the liquid is no longer in ebullition. The fat, which collects as an oily layer on the surface of the hot water, is continually removed by means of a shallow spoon, and collected in a high, narrow vessel. The residue in the boiling vessel—the feet freed from fat—is disposed of to glue manufacturers. On long standing, the neat's-foot oil separates on the surface of the water in this vessel as a clear oily layer, which is poured off from the water, and brought into small bottles of white glass, which are exposed to direct sunlight in order to bleach the fat. According to R. Brunner (*Die Fabrikation der Schmiermittel*, A. Hartleben, 1897), the bleaching is more rapid if a sheet of violet glass be placed in front of the bottle, for violet light has the most powerful action. The finest product is that which has been separated by freezing. The neat's-foot oil is, with this object, exposed to the intense cold of winter, and the residual liquid portion separated in the cold from the solid portion by fine linen cloth. In France, sheep's feet are first immersed in water heated to 75–80° C. for about twenty minutes, when wool and hoofs may easily be removed by hand; the feet are then boiled with steam. Four hundred sheep's feet give 1–2 litres of fat (0·22–0·44 gal.) of 0·915 specific gravity.

Recovery of Bone Fat.—Bones are not spoiled for the purposes of glue or bone-black making by the removal of the fat. The fresh bones are brought into a pan and covered with water, which is very slowly heated to boiling. The boiling must be continued for several hours, and the mixture

then left at rest. After five to six hours the crude fat has collected on the surface; it is removed, whilst still fluid, into a wooden pot lined with sheet-lead.

The best refining agent for this fat appears to be the so-called *aqua regia*, which is obtained by making a mixture in a glass vessel of one volume of white nitric acid and four volumes of strong hydrochloric acid, and allowing it to stand until it has acquired a reddish yellow colour and evolves a penetrating smell of chlorine. Not more *aqua regia* should be made than can be used within a few days, since it soon loses its activity. To 100 parts of fat contained in the above wooden vessel is added 1–1·5 part of *aqua regia*, which is mixed with the fat by prolonged stirring, and remains in contact with it for some hours. When the bleaching is finished, warm water is added, and the acid then run off carefully through a plug-hole at the bottom of the tank. The fat is again mixed with warm water, well stirred, and the water drawn off. This washing with warm water must be repeated until the last trace of acid is removed, *i.e.* until the water does not act on blue litmus paper. It should also be mentioned that the lead sheets, with which the vessel is lined, are at first strongly attacked. The white coating produced on the lead—lead chloride—should not be removed, since it protects the lead beneath from the further action of the acid in the same manner as a varnish.

When older bones are used, the above method for obtaining the fat must be somewhat altered. The bones are boiled with water, as above described, a brown fat of unpleasant odour being obtained, which is drawn off into wooden vats and allowed to stand for several days. During the slow cooling, a granular greyish-white mass separates, upon which a brownish yellow oil swims. The solid portion may be used in the manufacture of ordinary soap, the liquid in the preparation of lubricants. The fluid portion of the bone fat is bleached with *aqua regia*, as above described. The older the bones, the larger is the quantity of *aqua regia* required. The addition of any larger quantity than mentioned above must be very cautiously made; after 1 per cent. has been added, each fresh addition must not be more than 0·25 per cent.

Finally, in order to free the bone fat from the admixed oleïc acid, it is treated with about 10 per cent. of its weight of caustic lime, which is slaked with water immediately before use, and the resulting milk of lime stirred with the fat and then allowed to settle. The oleïc acid is thereby completely removed by the lime. According to Brunner's experiments, the oleïc acid may also be removed by an addition of litharge (lead oxide), especially if the fat be warmed before the introduction of the litharge. The lead oleate, which is produced, remains mixed with the fat, and converts it into a mass of the consistency of hard salve, which may be employed as a very useful lubricant for waggon axles.

A patent in 1873 by F. Sebor, of Prague, which was inaccessible to the author, related to the utilisation of the by-products of the manufacture of bone-black. In the only communication accessible to the author (*Dingler's Journ.*, 1873), Sebor states that not only had most of the bone-black works in Austria already adopted his process, but that many sugar-works also had introduced it.

Sebor summarises the advantages of his process as follows:—(1) A faultless and unadulterated "char"; (2) the production of an illuminating gas of excellent quality, since it consists mainly of oil gas; (3) the production of ammonium carbonate, which may either be converted into ammonia or another ammonium salt, or utilised in the form of ammonium sulphate as a very valuable fertiliser. The cost of producing "char" is reduced to a minimum by the utilisation of these by-products of bone-burning.

Recovery of Oil from the Cleaning Cloths of Machinery.—The most suitable method for the recovery of the oil is founded on the fact that all the substances used as lubricants are soluble in petroleum ether and benzene. The lubricant is dissolved in one of these solvents, and obtained by evaporation of the latter. The solvent is also regained, and may again be used for cleaning a fresh quantity of cloths.

The used cleaning cloths are squeezed fairly tight into a sheet-iron cylinder, provided with a tap at the bottom, and with an air-tight lid. Before the lid is put on, so

much petroleum ether or benzene is poured over the cloths that, after they are saturated, the solvent quite covers them. The lid is then put in place, and the vessel left at rest for twelve hours. The tap on a pipe about two yards long, attached to the lid, is then opened; water is run in through this pipe, and the lower tap opened. The solution of the solvent runs off; water is added through the pipe until nothing but pure water runs away below.

The solution of the lubricant is then brought into a distilling apparatus, the still of which stands in a pan of water. The water is then heated to boiling. Since both the solvents mentioned have a boiling-point below that of water, they evaporate completely, and can be regained by cooling the vapours. The lubricant, which was dissolved from the cloths, remains in the still in a fluid condition.

In order to renovate cleaning cloths saturated with fatty lubricants, the method of saponification may be employed. The cloths are brought into a pan, weak caustic soda solution poured over and heated to boiling. From time to time a few cloths are taken out of the liquid to see whether all the fat is yet saponified. When this is the case, the cloths are removed from the liquid, and boiling continued, when an ordinary soap is obtained. Cloths saturated with mineral oils or paraffin cannot be cleaned in this manner, but must be treated with the above-mentioned solvents.

Utilisation of the Residues of Wool-Washing.—Into the fatty waters, which are contained in large masonry tanks, sulphurous acid enters straight from the furnaces in which the sulphur is burnt (Chaudet, *Muster-Zeit.*, 1873). After the mass has become acid, it is allowed to stand for twenty-four hours. The tank then contains three separate layers,—the top consisting of impure fat, the bottom of an earthy deposit, whilst the middle layer contains the sulphites of soda, potash, and ammonia. This middle layer is concentrated by evaporation, and the residue burnt in a furnace. The sulphites are then converted into sulphates, which are obtained by solution and crystallisation. The upper layer is warmed and pressed in bags, in order to obtain the fatty acids.

CHAPTER XIV

FISH WASTE

Utilisation of Fish Scales.—In the year 1874 a method was discovered, by which fish scales, as such, might be used in the production of ornaments, artificial flowers, inlaid work, and the like. Layers of fish scales, bound together by any binding medium, are now better replaced by mica waste, which is more easily coloured and applied. E. and J. Huebner, of Newark, in 1874, obtained a patent for the following process:—The fish scales are cleaned in a suitable manner, until they appear transparent and horny. The large scales of fresh fish are the most suitable; old scales are useless, since they have lost their elasticity and transparency. In the process of Huebner the fresh scales are first treated for twenty-four hours with clean salt water, in order to loosen and remove partially the upper layers. They are then washed in distilled or clean rain-water, which is renewed every two to three hours. This is done five or six times. The scales are then separately and carefully rubbed with a fine linen cloth, gently squeezed in a press to remove moisture, and finally are laid for an hour in spirit, and again, as before, rubbed and pressed until dry. They have now the appearance of mother-of-pearl, and are very elastic and durable. They may either be used without further treatment, or may be coloured as required.

Fish scales are used to prepare "pearl essence," or "fish-scale essence." The dace, *leuciscus alburnus*, one of the commonest fish in our waters, is washed with water to remove any adherent dirt, and then carefully scaled, care being taken to remove scales alone, and not contaminate them with pieces of skin or blood. The scales are carefully collected and kept in a vessel of water, until a sufficient

number for the further treatment has been obtained. It is necessary to remove the scales from almost 40,000 dace, in order to obtain sufficient to prepare one kilo. of pearl essence, which quantity is, however, sufficient to make many thousands of artificial pearls. The vessel in which the scales are collected is best placed in an airy loft, since the scales very soon decompose, and then give off a most unpleasant odour. In order to avoid this inconvenience the following process is adopted:—The scales are covered, not with water, but with a solution of salicylic acid obtained by dissolving three grams of the acid in one litre of water (3 in 1000). The salicylic acid is tied up in a little linen bag, which is suspended in the water, so that the acid gradually dissolves. By using this simple precaution the collecting vessel for the scales may be allowed to stand in the summer without the least injury. When a sufficient quantity of scales has been collected, the liquid above them is allowed to run off, and a portion of the mass of scales transferred to a large porcelain mortar, in which they are ground for a long time with a flat pestle. In consequence of the grinding, those particles are removed from the scales which are the cause of their silvery appearance. After long rubbing, the mass in the mortar is mixed with water, and the whole poured on a closely-woven linen cloth stretched over a tub.

The colouring matter of the scales, which is suspended in the water in the finest state of division, penetrates the cloth together with the water. The scales are again ground and mixed with water, when a second quantity of the colouring matter is obtained. After the latter is entirely collected in the tub, the liquid is well stirred up, left at rest for some time, and then drawn off into glass bottles, in which it is allowed to stand until the colouring matter has completely settled to the bottom. The coarser portion of the colouring matter remaining in the tub is again stirred up with water, and allowed to settle.

The water above the colouring matter is carefully poured off. and the latter finally collected in one bottle. In the moist state it has a silver-white colour and a high glitter; on drying, a soft silver-grey powder is obtained. This "fish-

scale essence" can be kept for a long time unaltered under a solution of salicylic acid.

The mass with which the pearls are filled is prepared in the following manner:—Completely colourless gelatine is swollen in an aqueous solution of salicylic acid; the liquid is then poured off, and the gelatine melted to a clear liquid by cautious heating in a porcelain dish. The fish-scale essence is now introduced, and most intimately mixed with the gelatine. No definite figures can be given for the quantities of gelatine solution and fish-scale essence; the rule is to take no more of the latter than is absolutely necessary, because it is a costly substance. Thus no more of the "essence" is taken than is sufficient to cover the mass after it is introduced into the glass pearl.

The mixture is kept melted on a water-bath, and introduced into the glass pearls by sucking it up into a glass tube drawn out to a point, allowing a drop to fall inside the glass pearl, and turning the latter until the inner surface appears to be quite covered by the pearl essence.

In order to prevent the solidified mass from becoming detached from the glass wall, and to give the pearls a greater weight, they are filled with melted wax or a mixture of wax and paraffin. Pear-shaped pearls, which have only one opening, are filled with wax, and the opening closed by a small drop of melted pearl essence.

J. Loreau, at his works at Kernevel, near L'Orient, uses fish in the preparation of sardines in oil. A large quantity of waste is produced, consisting of heads, bones, entrails, bloody salt liquors, etc. This waste is collected, drained, heated in a pan and pressed; the cakes are dried and ground, when a fertiliser is produced. If treated with sulphuric acid, this fish guano would be very suitable for beetroot. (*Bull. Soc. Encouragement*, 1877; *Dingler's Journ.*, 227, p. 511).

Substitute for Isinglass, also Gelatine and Glue from Fishes and Fish Waste.—A substitute for isinglass, also for gelatine and glue, is prepared by C. A. Sahlström, of Stockholm, according to his patent, from fish and fish waste, by treatment with bleaching powder, potassium permanganate, and nitrous and sulphurous gases.

For this purpose the fishes, or portions of fishes, are first well washed in fresh water, and then left for from three to four hours in a solution of about 85 grms. of bleaching powder in 25–30 litres of water (1 in 300 to 1 in 350). After washing, they are treated with a solution of about 5 grms. of potassium permanganate in 25–30 litres of water (1 in 5000–6000), and then exposed to the action of the nitrous gases produced by heating 300–400 grms. of nitric acid for every 40 kilos. of raw material (about 1 per cent.). This gas may be first absorbed by water, as in the manufacture of sugar; or sulphur dioxide may be used instead of nitrous gases; the former would be obtained by burning about 200 grms. of sulphur for every 40 kilos. of raw material (about 0·5 per cent.).

The material, after this treatment, is washed; those portions intended for the production of the isinglass substitute are freed from their outer skins and dried and pressed at a gentle heat. The portions destined to produce gelatine or glue are, on the contrary, exposed to a temperature of 40–50° C. for from ten to twelve hours, by which the material is mainly dissolved. The mass is then forced through a strainer or sieve, allowed to stand for some hours, and finally dried, as is usual in the manufacture of glue or gelatine.

CHAPTER XV

CALAMINE SLIMES

Utilisation of the Residues.—Prof. Schwarz proposes extraction with hydrochloric acid, as a means for obtaining zinc from calamine slime (*Dingler's Journ.*, 218, p. 212). When the slightly roasted calamine is mixed with exactly the proper quantity of very dilute sulphuric acid required to saturate the zinc oxide, the latter is chiefly dissolved. When a calamine slime containing 9·5 per cent. of zinc oxide is lightly roasted, and then mixed with the quantity of 2½ per cent. hydrochloric acid equivalent to the zinc oxide, 5·6 per cent. of zinc oxide is dissolved. When the residue was again treated, the percentage of zinc was reduced to 1·5.

For 40 parts, by weight, of zinc oxide, or 32 of zinc, 73 parts of anhydrous hydrochloric acid are required, or about 243 parts of strong hydrochloric acid of 30 per cent. strength. Thus 100 kilos. of zinc. require 777·6 kilos. of hydrochloric acid. If the crude hydrochloric acid at the works cost 2·5 marks per 100 kilos. (about 1s. 3d. per cwt.), 100 kilos. of zinc would require acid worth 19·44 marks, which, with zinc at a price of 48 marks (24s. per cwt.), would leave a possibility of profit. There would be in addition the cost of precipitating by milk of lime, drying, and reducing to metallic zinc, although the latter, in view of the fine state of division of the precipitated zinc oxide, would take place readily and with a small consumption of fuel.

CHAPTER XVI

TANNERY WASTE

Utilisation of the Waste as a Fertiliser.—Opinions have been expressed against the use of spent tan bark as a fertiliser on account of the tannin it contains, which might injuriously affect cultivated plants. Still it may be remembered that all barks used for tanning purposes are completely extracted. In an article in the *Mark Lane Express* the above opinion was pertinently refuted. The tanning process, it is there said, gives rise to a quantity of waste and residues, which may very well have unrestrained use in agriculture—in fact, to a much greater extent than was formerly supposed. These residues are divided naturally into animal and non-animal. Agriculture can with advantage employ only those residues of each kind which cannot in any other manner be made useful, since it cannot pay the same price for its fertilisers as another branch of industry for a material out of which something useful can be directly produced. The animal waste of tanneries is much richer in manurial substances than the non-animal. In tanneries the fresh raw hides are first subjected to the prolonged action of milk of lime; they then undergo two operations, which determine the value of the tannery waste. In the first place, the hair is removed from the outside of the hide, and then remains for a long time in contact with lime. The second manipulation consists in removing the adherent flesh from the inner side of the hide, and also the thin cuticle. These waste matters are mixed together, and lie in heaps. They contain on an average about 75·5 per cent. of water and 24·5 per cent. of dry matter; the latter is composed of 84·5 per cent. of organic constituents and 15·5 per cent. of mineral substances. In 100 parts of the mineral matter are contained 3·25 parts of silicates,

17·5 parts of calcium phosphate, 69 parts of calcium carbonate, and about 10 parts of other salts. The mean proportion of nitrogen is about 7 per cent. Generally these heaps are from two to three months in collecting, during which time they lose about one-fifth of their volume of water and three-tenths of their nitrogen. This loss is due to the rapid decomposition of the animal matter under the action of the lime. This fertiliser has then, according to present prices of its proximate constituents, a value of 24–40 pfennigs. In strawberry plantations the soil is covered by a layer of tan bark about two inches deep. The fruit has then a clean bed, and the snails, the chief enemies of the strawberry, are kept off.

The vegetable portion of this tannery waste is a result of the tanning, which is only obtained after the hides have undergone their first treatment. This process is accomplished by using the bark of oaks or other trees rich in tannin, which is finely ground or powdered, and then put in alternate layers with the hides in the tan pits, water being then run in to dissolve the tannic acid.

The loose and fibrous nature of spent tan bark leads to the experiment of using it in place of *straw for litter*. When so used, it absorbs double its weight of liquid; it is advisable for this purpose to use it together with straw. It is not heavy, and may therefore readily be carried about. Tan bark, however, always still contains a certain percentage of tannin, and it is recommended to use it in combination with lime, phosphates, or ashes, materials which at the same time hasten its decay. In combination with animal excrement, tan bark accordingly forms a valuable fertiliser. In gardens it retards in the most marked manner the evaporation and drying of the soil.

Finally, there is known the utilisation of bark to form *bark cakes*, which are made in a press and used as fuel. Tan bark is also used, on account of its elasticity, for covering the floors of gymnasia, etc.

The Waste Waters of Tanneries in their Application as Valuable Fertilisers is advanced by J. S. Schultz in his *Leather Manufacture*. We follow here the views

laid down by Schultz, as communicated to the *Gerberzeitung* by F. Kathreiner, of Munich.

The water from softening, as also the lime-pits and tan-pits, which have become useless, and the wash waters from the scouring and cleansing operations, are all collected in a large tank placed below the level of the tannery, so that all these valuable liquid fertilisers may be collected without pumping. This reservoir may be at some distance from the position when the washing and scraping operations are performed; in fact, it is an advantage if it be several hundred feet away rather than near at hand. It should be of sufficient size to contain, not only the waste waters, but also the solid matters which come from the sweepings and cuttings on the floors of the washing and drying places. These waste waters are so valuable that the author could see how a small tanner could fertilise 100 acres with the waste of a tannery treating 5000 hides. Exhausted bark, fine cuttings and scrapings, and even earth, may be brought into this reservoir, in order to take up the ammonia, and then be added to the soil with great profit.

Waste, consisting of Pieces of Untanned Hide, and of "tawed" leather, may be used in the preparation of the finer kinds of glue. On account of the differences in the material, the waste from untanned hides and from leather is collected in separate vessels. The waste of "tawed" leather, if it has been sufficiently dried by exposure in thin layers, may be stored, without further treatment, in any vessel until a sufficient quantity has been collected. The waste from untanned hides may be treated in the same manner, but it is not dried, since the skins, being saturated with water, are precisely in the condition from which they can easily be converted into glue. The glue maker has therefore again to soften the dried waste, and labour is thus lost. The hide waste is therefore left in the swollen state, care being taken that it does not decompose. This is done by keeping it in a tub under clear lime water, which must always cover the cuttings and also contain sufficient lime. Fresh lime water is therefore added from time to time. When the tub containing the cuttings is placed in a cool room, they may be kept for four to six weeks

without decomposition; if they are then to be worked up for glue, the lime water is run off and the hide washed several times with pure water.

It is still more convenient to keep the hide cuttings in a weak solution of carbolic acid—1 in 1000. When they are to be used, they are taken out of this solution, allowed to drain, and washed with water. The carbolic acid solution may be used to preserve fresh quantities of hide.

Hide clippings may be converted into glue without further treatment. The clippings of "tawed" leather are best treated repeatedly with cold water, and then with hot water, in order to "untan" them and, as far as possible, to obtain the original hide. Clippings of chamois leather can only with far more difficulty be converted into glue, since in them the tanning substance is far more firmly combined than in "tawed" leather. It is best to boil these clippings with weak caustic liquors, by which they are at least partially untanned.

In order to convert into glue the clippings of leather tanned with alumina soaps, they must first be boiled in a liquid containing an acid, such as sulphuric, in order to decompose the insoluble soaps. The clippings are then squeezed and boiled with a weak caustic lye, in order to convert into the soluble condition the free fatty acids now present.

CHAPTER XVII

GOLD AND SILVER WASTE

Recovery of Gold from Liquids containing the Metal.—Boettger (*Dingler's Journ.*, 213, p. 257; *D. Ind.-Zeit.*, 1874, p. 296) brings the liquid to the boil in porcelain vessels, adds a solution of stannous oxide in caustic soda, and continues the boiling until all the gold—in combination with tin—has separated as a fine, intensely black precipitate, which is pressed and then dissolved in *aqua regia.* The liquid thus obtained is a solution of gold and stannic chlorides; it is carefully evaporated, diluted with distilled water, a sufficient quantity of sodium potassium tartrate (Rochelle salt) added, and the mixture warmed, when every trace of gold separates as a very soft brownish powder, whilst the tin remains dissolved.

Recovery of Gold from Galvanic Baths.—According to Plage (*Industriebl.*, 1878, p. 190), a porous cell containing a solution of salt is placed in the gold solution; inside the cell is placed a zinc rod, which is connected by a brass wire to a thin brass plate immersed in the gold solution. In three to four weeks, after once or twice renewing the brass plate, the zinc rod is dissolved, and all the gold is precipitated on the brass. The latter is dissolved in nitric acid in a porcelain dish, the gold is washed, dried, and may finally be melted with saltpetre in a Hessian crucible.

Recovery of the Gold from old Photographic Toning Baths.—F. Haugk (*Photog. Arch.*, 1877, p. 6) filters old gold baths into a white glass bottle, adds excess of sodium carbonate, and then an alcoholic solution of magenta, drop by drop, until the liquid has become raspberry-red. If the flask is now placed in a light window, the gold is entirely precipitated in six to eight hours, so that the liquid may be

poured off. When a sufficient quantity of the precipitate has collected by repeating this process, it is well washed and brought on to a filter, which is burnt, and the gold then dissolved in excess of *aqua regia.* By evaporation of the solution, gold chloride is obtained, which may at once be again used for photographic purposes.

Collection, Reduction, and Purification of Silver and Gold Residues.—Complete instructions for these operations have been given by J. Krüger (*Neueste Erfindungen*, 1878, p. 194), taking all photographic waste of this description into account, and showing exactly how the highest yield may be obtained from all residues, and the processes best conducted with the ordinary technical appliances.

1. *Liquids which contain Noble Metals.*—If we follow the numerous photographic processes required by the collodion and silver printing process, we obtain the following solutions:—(*a*) Old, exhausted, and useless silver baths; (*b*) developing and intensifying solutions; (*c*) fixing baths, and (*d*) the wash waters of these operations; (*e*) the wash water of the silver prints; (*f*) old gold baths; (*g*) old fixing baths; and (*h*) the wash waters after fixing. At first sight it is seen that all these solutions differ principally by containing the silver in quite different compounds and forms—partly in solution, partly suspended, partly reduced, partly dissolved as double compounds. On this account it is advisable only to mix liquids of the same kind, and not, as is often done, to bring all together. Thus, all old silver baths should remain by themselves, as also the intensifying liquids and the solutions and wash waters containing sodium hyposulphite; for by mixing, not only is the separation of the silver made more difficult, but also a considerable loss is introduced. Accordingly, we shall consider these liquids in turn, in order to learn how they may most simply, cheaply, and easily be worked up for silver.

(*a*) *Old Silver Baths.*—The silver bath used in the negative process finally contains, when it is completely exhausted and saturated with silver iodide, the silver in the form of mixed compounds; it is principally the compound of silver iodide and nitrate ($AgNO_3 + AgI$) which makes the reduction, or

rather the recovery, difficult. When I hear and see that many photographers adopt an easy course by simply evaporating these baths to a strength of one part of silver salt to ten parts of water, and at once using this solution to silver albumin paper, I cannot repress a shudder. I take this opportunity of giving an earnest warning against this practice; for I thought it was universally known that the silver iodide, which is dissolved in the solution, and is thus conveyed to the albumin paper, does not give brilliant whites and pure tones. It should not be overlooked that even by a long-continued fusion of the residue of salts produced from these old negative silver baths, the silver iodide therein can neither be separated nor reduced, but rather remains, and can only be destroyed by the decomposition of the double compound. The most general method for separating all the silver salt contained in these baths is to add to them common salt or hydrochloric acid, so long as a precipitate is produced; the silver is thus precipitated as chloride. This precipitation occurs completely under all circumstances, and hence no objection could be taken to the process, if the silver chloride obtained were not more difficult to reduce and smelt than another silver salt—the carbonate. For if, in place of common salt, pure soda (sodium carbonate) is used for precipitating the silver, then silver carbonate is obtained, which has many advantages for our purpose of reduction: it deposits more rapidly, and is more easily and quickly reduced, since it does not froth up so much as silver chloride in the fusion. The same process is used with the soda solution as with the common salt solution; the mixture is vigorously stirred, allowed to clear, and then more precipitant added so long as a precipitate is produced. It might be objected that sodium chloride only precipitates silver, whilst soda also throws down other metals. But this is not material, since in the silver bath neither iron nor copper nor similar metals are to be feared; and the small quantities of cadmium derived from the collodion are either taken up by the slag in the fusion or burnt away. The silver carbonate thus obtained is a pale yellow powder, easily reduced; it requires in the fusion only a small addition of sodium carbonate as flux, in order rapidly to obtain a pure regulus.

(*b*) *The Developing and Intensifying Liquids.*—These liquids are more or less dark in colour, according as green vitriol alone, or green vitriol and pyrogallic acid, are employed; the precipitate which forms in the liquid is also of varying purity. It always consists essentially of reduced silver, which is precipitated more or less pale, according to the purity. It is collected, washed, dried, and fused with a little soda alone, when it gives a solid lump of metallic silver. Every photographer who himself carefully collects these liquids will be well rewarded by the success, for the profit is easily obtained, and easily surpasses the trouble. This precipitate may be smelted together with the precipitate of silver carbonate obtained from the old silver baths.

(*c*) *Fixing Solutions and their Wash Waters.*—In view of the difference between the fixing salts—potassium cyanide and sodium hyposulphite—it is, in the first place, important to keep the two separate, and also the corresponding wash waters. In the case of potassium cyanide, which is mostly used for negatives, two methods may be adopted in order to recover the silver: the liquid may either be evaporated to dryness, and the residue ignited, or it may be treated with pure hydrochloric acid, so long as a precipitate is formed. On account of the extremely poisonous hydrocyanic acid gas evolved in the latter operation, it must be conducted in the open air, or under a chimney with a good draught. The potassium cyanide is then decomposed, potassium chloride is formed, and the dissolved silver is precipitated. In evaporating—which may be done in summer in the air—in a hot, draughty room the potassium cyanide decomposes spontaneously; finally, a formate is produced, and there remains a dark brown mass of salts, from which all the silver may easily be obtained by fusion. It is highly to be recommended to allow all potassium cyanide solutions, from which silver is to be recovered, to decompose spontaneously in the air, by which they lose their poisonous properties and separate a large portion of the silver.

Old hyposulphite baths and their wash waters are generally collected and sold, but naturally something must then be lost, and it is at least advisable to know the amount

of silver in the liquid. This is simply found: exactly 100 grms. are measured off, diluted with 200–300 grms. of water, and a strong potassium iodide solution added so long as a precipitate is formed. This precipitate is silver iodide; after washing and drying, it is weighed and the corresponding quantity of pure silver calculated. This may be done by means of the following table:—

2·176	parts of silver iodide	= 1·000	part of metallic silver.		
1·386	„ „	= 0·635	„	„	
1·637	„ „	= 0·752	„	„	
1·000	„ „	= 0·459	„	„	
1·250	„ „	= 0·574	„	„	
1·753	„ „	= 0·806	„	„	
1·813	„ „	= 0·871	„	„	

This method depends on the insolubility of silver iodide in weak solutions of sodium hyposulphite. As a rule, however, it will be found that, on long standing, sodium hyposulphite separates a portion of the silver chloride dissolved in it. Silver sulphide is deposited both at the bottom, where it forms a black mud, and on the sides of the vessel, which must be remembered in emptying the contents. The silver sulphide, which is not an insignificant amount in old hyposulphite baths long exposed to the air, is calculated to the corresponding quantity of silver by the following table:—

Silver Sulphide. Parts by weight.		Metallic Silver. Parts by weight.
1·148	=	1·000
0·729	=	0·635
0·864	=	0·752
0·528	=	0·459
0·669	=	0·564
0·926	=	0·806
1·000	=	0·871

The method, recommended by several writers, of decomposing these old hyposulphite baths by hydrochloric acid, in order to obtain the dissolved silver as silver sulphide, is not considered advisable by the author, for, on the one hand, the

sulphuretted hydrogen gas which is evolved is extremely dangerous to health, and very harmful to all photographic work (on which account the operation must be performed at a distance from the studio and in the open air), and, on the other hand, so much sulphur is separated that its removal necessitates a separate operation, and makes much more difficult the further treatment of the precipitate. Such precipitates containing sulphur can only be reduced by fusion, and since in this case the addition of pure soda or other salts does not suffice, iron must be added to the mixture in the form of wire or nails, or as filings, so that sulphide of iron may be formed and the difficultly reducible silver sulphide obtained pure. Under all conditions the utilisation of these liquids by the method just mentioned is as complicated as it is unpleasant, hence sale is the more advisable method of disposal. In the author's many years' practice he has endeavoured, in face of these obstacles, to discover a simpler process, which would be neither too costly nor too complicated for the photographer. The best results may be obtained by the process next described.

A large wooden vessel, in which all old hyposulphite baths and wash waters are collected, is provided with a series of plug-holes, in a vertical line, at distances 5 cms. (2 ins.) apart, from the top to immediately above the bottom. All the holes are closed by well-fitting corks. A broad clean strip of zinc, wound round with a fairly strong copper wire, and long enough to project out of the liquid, is brought into this vessel. When this apparatus has stood for some time in the liquid, the latter will be found to have become quite colourless, whilst the surfaces of the metals are covered by a grey metallic powder, which can be readily loosened and wiped off. This consists of metallic silver, which partially sinks to the bottom and partially adheres to the copper and zinc; in quantity it is equivalent to the metals dissolved. This precipitated silver is certainly not chemically pure; it contains zinc and copper, but their presence offers no difficulty in the subsequent fusion process. The separation of the silver from these liquids by phosphorus is very efficient, but costly; on the large scale it would be difficult, if not im-

possible, of execution. The addition of green vitriol, which the author also tried, is not without effect, but the silver precipitate then appears deep black in colour and contains sulphur and sulphide of iron. Copper sulphate (blue vitriol) behaves in the same manner. The organic acids, such as gallic and pyrogallic, are partly inefficient, partly too expensive. More certain and less costly, though large vessels and much water are required, is the precipitation of the silver by potassium iodide, as given above, since weak decomposed hyposulphite solution cannot dissolve silver iodide.

The utilisation of old liquids containing gold will be considered afterwards. In the next place, we shall see how the silver precipitates are to be further treated; but before going on to the necessary processes of fusion or smelting, the methods for treating solid residues containing silver, before fusion, are given.

2. *Solid Residues containing Silver.* — Under this heading come—(*a*) old silver filters; (*b*) old enclosures in the plate box, also the paper wrappings on the prepared plates, etc.; (*c*) clippings from the prints. In order to reduce the volume of these papers, they are first burnt, in which operation it is advisable to sprinkle fine, dry, powdered saltpetre over the glowing mass, by which a slight explosion is caused and a more complete combustion of the carbonaceous matter of the paper (and, in albumin papers, of the albumin) is effected. It not rarely happens in this operation that the silver is obtained in the form of very fine minute globules of metal. This is not the object of the process, but simply, by diminishing the volume of the material, to save space, and thence expense and inconvenience. These ashes may then be at once mixed with the silver precipitates obtained from the various liquids, so that they may be reduced together. With this we reach the second part of our process—the smelting out of the silver. The author does not think it necessary to state to what extent this operation may be performed by the photographer himself, but simply states that many are of the opinion that this smelting may be accomplished in an ordinary iron stove, and even maintain

that the best results are then obtained. That this is not altogether unwarranted will appear in the following pages, but it must be at once understood that this troublesome and dangerous operation has not for its object the obtaining of some reduced silver as a regulus, but to regain the whole of the silver united in one lump, and that with the least possible loss.

Smelting out the Silver.—In whatever form the silver is collected, it must, in the first place, be thoroughly dried, either by spreading out and leaving in the air, or, in case that is not sufficient, by artificial heat. Care must be taken to see that this drying is thorough, for if the residues are thrown into the heated crucible while still moist, it would almost certainly crack, which is to be avoided. In every smelting operation two things are required—the complete reduction of the metal and its union to one lump, on which account certain substances require to be added. This addition consists almost always of an alkali salt, a carbonate, nitrate or chloride, *i.e.* a compound, which can both separate the silver compound to be reduced and also itself readily fuse, so that the silver, produced in small globules, may separate completely, sink to the bottom of the fluid mass, and there collect. If we have to do with pure silver masses, such as result from the developing and intensifying solutions, and also with silver carbonate, the smelting process proceeds calmly, exactly, and rapidly, whilst silver chloride, and silver sulphide to a greater extent, make the process difficult.

The fluxes used and proposed for these operations are as little of the same nature as is the residuum or the waste resulting from the various photographic operations. The following salts are chiefly employed for this purpose: potassium and sodium carbonates, common salt (sodium chloride), sodium biborate, and also the so-called "black flux," a mixture of caustic potash, potassium carbonate, potassium tartrate, and carbon. These salts differ essentially in their action on the metals to be reduced. The alkaline carbonates are reducing agents and also excellent fluxes. They take up the chlorine, bromine, sulphur, etc., giving off carbon dioxide and forming the corresponding potassium

and sodium salts, which then form a light flux, which dissolves accidental impurities, such as iron, copper, lead, and also calcium salts, and separates completely purified silver. They are the most often used, and since it is desirable to combine the advantages of the potassium salt, which (as also the sodium salt) forms a readily fusible glass, with those of the sodium salt, which is better to handle since it can readily be converted into a dry powder, whilst the potassium salt is very deliquescent, frequently the two are mixed, two parts of sodium carbonate being well ground with three parts of potassium carbonate. The alkaline nitrates have an oxidising action on the base metals present, and also effect the complete combustion of carbon. Both are used in refining the noble metals, small quantities being added to the carbonates, in order to obtain purer gold and silver. The nitrates are not used alone. Borax acts in the same manner as the nitrates, yet not by oxidation, but by forming glassy fluxes with most of the base metals, *i.e.* it dissolves them, so that they are completely converted into a coloured glassy slag. It is thus equally useful in refining, and is also very valuable because it is a good flux, which soon brings the slags into a very fluid state, by which the separation of the noble metals is much facilitated. Borax is generally used towards the end of the smelting process, in order to increase the purity of the silver and gold and help them to melt together. Common salt, which is much valued by some, is only useful on account of its ready fusibility. It is not correct to say that common salt reduces silver chloride; it simply serves as a flux to fuse together the particles of silver, for which it is very suitable, because it is readily fluid, and under those conditions the silver can readily collect without any loss of the metal. For this purpose the author can recommend it from his own experience. Under the name of "black flux," a preparation is known which formerly was almost exclusively used in smelting the noble metals. As already mentioned, it consists essentially of potassium carbonate and organic carbon; it is obtained by mixing two parts of potassium bitartrate with one part of saltpetre and setting the mixture on fire by means of a red-hot coal. A slow explosion occurs, with the produc-

tion of much smoke; finally, when the mass is quite burnt and has cooled, a black carbonaceous mass remains—black flux. This mass is ground whilst still warm and at once brought into well-closed glass bottles, in order to prevent it from absorbing moisture from the air, which it rapidly does on account of its deliquescent nature. In addition to these salts, which, however, are always sufficient in a stove with a good draught and sufficient fuel, the addition has been recommended of rosin, sugar, and even soaps and syrup, as reducing agents, but the author does not agree with this advice, for what cannot be accomplished by the salts will never be attained by means of these easily decomposed organic matters.

We now come to the charging of the crucible. Melting crucibles are conical cylindrical vessels of fire-clay. The clays used for most vessels for industrial purposes are never of such a composition that they can be used for melting crucibles. It is necessary to give them the requisite resistance by certain additions. The reasons for this are twofold: to impart indifference towards the fused mass, and the property of not cracking or breaking. A clay which possesses these properties is that mined at Almrode, from which are obtained the Hessian crucibles used all over the world, by adding half of its weight of coarse quartz sand. In spite of their durability in the fire, these crucibles require a certain amount of care, since all substances cannot equally be fused in them. This relates to those compounds which are able to dissolve quartz (silicic acid) and thus form silicates. In this case the walls or the bottom of the crucible may be perforated, so that the fluid contents may drop or flow out. To these substances belong free alkalis, free bases, and metallic compounds which form glasses with silica, as, *e.g.*, litharge. To crucibles in which this substance is to be fused, in place of the quartz an addition is made of finely ground fireproof pottery. Other manufacturers take for this purpose also difficultly combustible carbon, such as graphite and coke. Another kind of crucible is the Passau crucible, made from one part of plastic clay from Schildorf, near Passau, with two to three parts of an impure graphite from the same district. These crucibles

may, without danger or the least alteration, be heated up to 150° Wedgewood. They resist changes of temperature very well, and have the particular advantage over all other crucibles of a similar kind that the inside is quite smooth, which is a great advantage and very important in smelting the noble metals, since then the fluid slag can be poured out clean and without sticking to the crucible sides. If this crucible has not the fame of the Hessian, it is because it is too dear for all those processes in which the melting vessel is broken after cooling at the end of each operation. In conclusion, it is to be noted that all crucibles soon become useless, they rarely can be used for any large number of fusions.

The crucible is filled in the following manner:—The silver ashes, residues of the developing process, and salts precipitated in the form of silver carbonate are, in a perfectly dry condition, well mixed with the additions, which for this purpose are—potassium carbonate three parts, sodium carbonate one part. If, after thirty minutes' heating, the mass is near fusion, one part of saltpetre is added. Equal parts of silver ashes and the flux are taken. The crucible should at first be at the most two-thirds full, and it is advisable to cover the contents with a thin layer of common salt. If pure silver salt residues are melted together without the ash constituents, a mixture of equal parts of common salt and potassium carbonate is sufficient. When the mass melts, a very little dry powdered saltpetre may be added. Old collodion films, with and without varnish, both before and after fixing, contain silver bromide and iodide, and are therefore less easily reduced, yet a mixture of potassium carbonate four parts, sodium carbonate one part, and sodium chloride one part, added in equal quantity, will effect the result if a fierce and regular fire be maintained.

Silver sulphide is the most difficult to reduce, yet the operation is easy if it is treated with equal parts of the following mixture:—sodium carbonate, 3 parts; common salt, 1 part; iron filings, ½ part. An excellent reducing agent for silver sulphide is potassium cyanide, of which also equal parts by weight are used. Potassium sulphocyanide is then produced, and chemically pure silver separated.

Silver chloride, which the author avoids when possible, may be reduced by the method given for collodion films, in which it is advisable to apply the common salt by sprinkling it into the crucible.

In all these mixtures care is to be taken that the separate parts are quite dry and that the crucible is at first only three-quarters full. After filling the crucible, it may be placed in the stove. In the first place, a piece of roofing tile, of the circumference of the crucible—but not greater, so that the draught is not restricted—is laid on the fire-grate. The crucible is then placed upon it, surrounded by wood, and the latter lighted. As soon as it is sufficiently ignited, the wood and the crucible are covered with charcoal and coke, care being taken that the fire is always close round and covering the crucible. A long iron poker is used to order the fire. All the fuel must be dry, the crucible always well surrounded and covered. When the first charge of the crucible is quite fused, small portions of the residues are taken, wrapped in a piece of paper, and the ball brought into the fluid mass. A slight frothing follows, but in a short time the contents of the crucible are again in quiet fusion. The addition of fresh portions is continued in the same manner until the whole is introduced. With some care and attention boiling-over need not be feared; if the mass rises considerably, it is stirred with a cold poker, when it at once falls. After the addition of the last portions, the fire is brought to its fullest heat, well stirred, new fuel added, and efforts made to reach the highest temperature. This can be done in half an hour.

When it is thought that the reduction is finished, the fused mass of salts is examined, both as to its appearance—since it must be at a white heat and quite fluid—and by means of an iron wire, which is plunged into the melted mass, and a test taken. The wire becomes covered by slag, which is examined with a lens, to see whether fine, scattered globules of silver are present in it. The slag should also be clear, and neither rough nor sandy, which would indicate that the mass is not yet sufficiently fluid to allow these impurities to settle. If the test shows any defects—and especially that the silver has not completely separated and united to one

mass—the fire must be increased, and the contents of the crucible again properly fused for some time, which is continued until a new test shows the requisite signs of complete reduction and collection of the silver. This result is occasionally attended by difficulties, and, in fact, always when the stove is not provided with a sufficiently good draught or the necessary dampers, for even with sufficient fuel the necessary heat cannot be attained without a rapid draught.

When the operation is successfully finished, the crucible is taken out with the tongs and placed on a heated stone slab. The silver may be removed either by pouring out or allowing to cool. If the silver is poured out, an iron vessel—say a mortar—is well greased with tallow, the fluid silver and slags poured slowly in, and, after completely cooling, the contents are knocked out, and the silver freed from adherent impurities.

If, on the other hand, the crucible is allowed to cool slowly, the silver adheres fast to it; the cold crucible is then finally broken, and the silver obtained as a rounded compact lump, free from slag. An attentive worker will always obtain a favourable result by carefully following these directions; but a certain amount of practice, and the knowledge thereby obtained, are of great advantage. A beginner should never throw away the slags without previously examining them for silver. This is done with the lens, and also, more certainly, by dissolving a small portion in hydrochloric acid, which does not attack metallic silver. If the slag is found permeated with silver, it must be again fused at a higher temperature.

Recovery and Smelting out of Gold.—All liquids which contain gold are collected, and, if alkaline, acidified with strong hydrochloric acid. Any precipitate produced is disregarded. To the acid liquid is then added a strong solution of ferrous sulphate, so long as it produces a precipitate. Excess is not injurious, but the liquid ought not to be very acid. After a short time a black precipitate of reduced gold settles to the bottom, and is carefully filtered off. If there is plenty of time, the precipitate may be allowed to deposit completely, and only the last portions filtered, after all the clear liquid has been poured off. The residue on the filter is many times

washed by pouring clean water over, and then dried. When this is done the filter is burnt at an open flame, the ash and its contents collected, and both mixed in a porcelain mortar with double the weight of pure dry (or effloresced) sodium carbonate; the mixture is then at once brought into a crucible of suitable size.

The crucible must have double the capacity of the mixture; it is covered by a second crucible, fitting into the top, and the two luted together by good clay. The little apparatus shown in Fig. 8 is then obtained, viz. the crucible *A*, containing the gold residues; the crucible *B*, serving as a cover; and the junction *C*, made tight by a layer of clay. The whole apparatus, as thus arranged, is placed on a small piece of stone in the fireplace, then surrounded by charcoal; as soon as the charcoal is burning strongly, the whole is covered with coals, and a strong white-heat maintained continuously. The process is finished after about two hours; the fire is then removed, the crucible allowed to cool slowly, and then opened, when the lump of gold is found at the bottom.

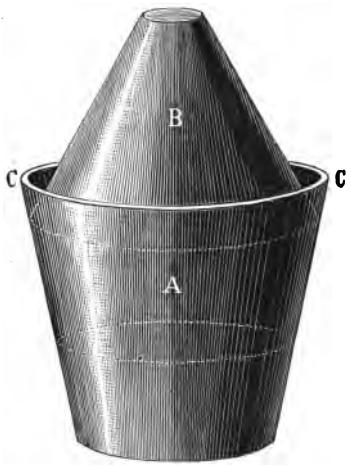

FIG. 8.

This is the simplest and safest method; it deserves preference because, if foaming and frothing should occur, no trace of metal can be lost. The little apparatus is also recommended for smelting silver residues.

Photographic Paper Refuse: Filters.—This waste is incinerated, and the ash treated with dilute nitric acid. The liquid is then filtered, and the silver precipitated by copper. The ash may also be digested with strong hydrochloric acid free from nitric acid, and the washed and dried residue fused with its own weight of soda and about 10–15 per cent. of saltpetre. The metal obtained is then dissolved in nitric

acid, when a residue containing gold may remain. This residue is dissolved in *aqua regia,* the solution diluted and precipitated by green vitriol; the gold obtained is washed, dried, and melted with saltpetre and borax.

In regard to the method just given, in which the silver is separated by copper, it is to be observed that in the Frankfort Gold and Silver Separating Works, the silver obtained in sulphuric acid solution is precipitated by iron instead of copper. The objections to this process are, the loss through the evolution of hydrogen, the simultaneous precipitation of copper, and the contamination of the silver by phosphorus, silica, etc., from the iron. The objections are, however, removed by allowing the silver sulphate solution, with the addition of water, to crystallise, and then reducing the crystals by scrap iron. The crystals are stirred with water, and iron gradually added, so that every trace of silver is precipitated, whilst copper remains in solution. The impurities contained in the iron go into the slags produced when the silver is fused, so that the latter is obtained more pure than when copper is employed. The principal advantages of this method are—economy in acid, since the mother liquors from the crystals again go into the dissolving vessel; avoidance of the purchase of copper, which is not well utilised as blue vitriol; economy in steam, since the solution becomes very hot spontaneously.

CHAPTER XVIII

INDIARUBBER AND CAOUTCHOUC WASTE

Utilisation of the Waste.—According to Grote, in Muspratt's *Chemie*, Goodyear grinds the waste from vulcanised caoutchouc in a rag-engine, mixes with it pure caoutchouc similarly divided, passes the mixture between heated rollers in order to obtain thorough incorporation, then adds a quantity of sulphur corresponding to the pure caoutchouc, and afterwards proceeds as in the manufacture of vulcanised caoutchouc.

Bacon disintegrates the waste of vulcanised or hardened caoutchouc or gutta-percha, then pours 10 kilos. of carbon bisulphide and 250 grms. of spirits of wine over 100 kilos. of the waste, and allows the whole to stand for two hours in closed vessels, when the mass will be found so soft that it can be ground and used again in the manufacture of new objects. The degree of softening can be regulated by the quantity of the carbon bisulphide mixture and the duration of the action.

Parkes (according to Grote in Muspratt's *Chemie*) boils 8–10 kilos. of the disintegrated waste in a strong solution of 20 kilos. of calcium chloride, until the gutta-percha or caoutchouc has become soft and two pieces can be readily united by kneading. The lumps taken out of the liquid are washed first in a hot alkaline liquor, then in hot water, after which, according to the statements of the patentee, they can again be worked up.

Newton desulphurises caoutchouc waste by softening it in camphine (distilled turpentine) for two to fourteen days, according to the extent of the vulcanisation. If much sulphur was used in vulcanising, or if lead compounds are present, it offers considerable resistance to the camphine. The treatment

is in every case continued until the waste is well softened throughout, when it is heated in a still at 65–71° C., adding camphine from time to time, so that the waste is always covered with liquid. It is convenient to add in this operation 15–25 kilos. of ether and 5 per cent. of spirits of wine, and then continue the heating until the waste has regained its natural condition, for which one to two hours are requisite; the whole is then dried at a gentle heat. The addition of spirits of wine prevents the residues from remaining sticky, and may be omitted if this is immaterial. The use of a still effects the recovery of the evaporated solvent.

Later, Dodge recommended to disintegrate the waste as completely as possible and then expose it to a temperature of 150° C. in a cylinder surrounded by another cylinder, steam being supplied to the space between, until it had become quite plastic, when it was mixed with fresh rubber mass, or to 25 kilos. of waste, 100 grms. of palm oil, 170 grms. of sulphur, and 1·5 kilo. of white lead, zinc white, magnesia or clay may be added, and the whole well kneaded.

A new and improved process for utilising the caoutchouc contained in the waste of caoutchouc goods, and for recovering the zinc present in them, is due to Burghardt, of Manchester, and has been patented in England. In this process the waste is thrown into a vessel of wood or lead, and covered with the requisite quantity of hydrochloric acid of a strength sufficient to destroy adherent cloth and fibres completely; the mixture is then boiled until the caoutchouc is freed from all fibres. The solution contains zinc and calcium chlorides, and occasionally also lead chloride; it is treated as described below. The vulcanised rubber is now brought into a vessel containing water, which is boiled until all the acid and zinc, calcium, or other chlorides are removed from the rubber. The caoutchouc thus purified is now dried, and mixed with sufficient spirit, coal-tar naphtha, wood-naphtha, petroleum, turpentine, carbon bisulphide, benzene, or other solvent for rubber, in a jacketed vessel of wood, copper, iron, or lead, the vessel closed, and the whole brought to a temperature of about 115° C. by means of steam, gas, or hot air, until the caoutchouc is dissolved or converted into a thick pasty

mass. When this is the case, the naphtha is distilled off (according to the *Chemiker-Zeitung*) at a temperature not exceeding 100° C. The above-mentioned solution is treated in a vessel with sodium bicarbonate, soda crystals, anhydrous sodium carbonate, ammonium carbonate, potassium carbonate, the carbonates of the alkaline earths, caustic soda, caustic lime or its solution, or caustic potash, in quantity sufficient to precipitate the zinc, calcium, or lead from their solutions. The precipitate is ground in a current of water, by which the calcium carbonate, which is specifically lighter than the zinc carbonate, is carried away. The zinc carbonate is either dried and sold as such, or dissolved in hydrochloric acid, sulphuric acid, etc., and sold as zinc chloride, sulphate, etc. The solution may also be concentrated to a suitable extent and used as a mordant for cotton.

Ludwig Heyer, a rubber manufacturer of Berlin, applies the following method **to the recovery of old vulcanised caoutchouc**:—Old discarded buffer rings of railway waggons are heated in the presence of steam. The sulphur distils off, the caoutchouc melts and flows into hot water, where it collects at the bottom of the vessel. The steam prevents the material from burning. The melted caoutchouc has acquired essentially different properties; it has become a tolerably fluid dark mass, which also remains liquid at the ordinary temperature, soon dries in the air, and has retained the property of being waterproof. On the other hand, it has lost the elasticity, at least in thick pieces. Heyer employs this fluid caoutchouc principally for preparing waterproof sheets for waggons, railway waggons, ships, etc. It may also be recommended as a waterproof varnish for ironwork. (*Chemiker-Zeitung*, 1880.)

Dankwerth and Köhler, of Petersburg, have patented a *process for the treatment of old or perished caoutchouc, in order to obtain from it oils, varnish, and materials for mixing with caoutchouc and other substances.* Old or perished caoutchouc is subjected to dry distillation in a retort by means of fire and superheated steam. Volatile constituents of the caoutchouc pass over, which, when condensed, form oils and a substance which has, after vulcanisation, to a great

extent the properties of natural caoutchouc. The lighter oils, distilling at 60-105° C, are separated from the heavier oils. The former serve directly for the preparation of varnish, the latter are mixed with hempseed, linseed, or other vegetable oils, and then converted into boiled oil by boiling or chemical action. The mass still to be subjected to vulcanisation is intimately mixed by means of rollers, and then 7–20 per cent. of sulphur added.

A very durable *varnish from old rubber waste* can be prepared for various purposes (Campe, *Oel- und Fettindustrie*, 1890; *Neuste Erfindungen und Erfahrungen*, 1890) by heating 2 kilos. of old rubber, 4 kilos. of colophony, 4 kilos. of boiled linseed oil, and 0·5 kilo. of sulphur in an iron pan until all are equally dissolved. When this is the case, whilst the liquid is still hot, 4 kilos. of rectified American spirits of turpentine are added, and then the liquid is diluted with about 10–12 kilos. of boiled linseed oil. The varnish may be coloured with earth pigments as desired, or left in its original state. When uncoloured, the varnish has a honey-yellow colour similar to a dark copal varnish. It is particularly recommended for varnishing the lead roofs of bleaching-powder chambers; its resistance to free chlorine has been tested for years. The varnish is very brilliant; when good boiled oil is used in its preparation, it dries within several days.

Artificial caoutchouc is made in the following manner:—In an iron pan about 10 lb. of sulphur or flowers of sulphur are heated with 20 lb. of rape oil, with constant stirring, until the sulphur is melted and a point is reached at which the mass swells up. At this instant the mass must be rapidly poured out, either into a mould dusted with any kind of flour, or on to stone plates wetted with water. The mass solidifies, and has similar properties to caoutchouc; it is soluble in linseed oil. The mass may also be prepared from linseed oil, but less sulphur must then be taken. The pan should only be half filled, otherwise the mass would boil over.

CHAPTER XIX

BY-PRODUCTS IN THE MANUFACTURE OF ROSIN OIL

Recovery of Acetate of Lime from the Acetic Water of the Rosin Distillation (*Die Harze und ihre Producte*, G. Thenius. Vienna, Hartleben, 1895).—The acetic liquor is neutralised in large wooden vessels, when acetate of lime is formed, and a brown insoluble compound of lime with resinous matters separates at the surface and is removed. The clear liquid, obtained by filtering, is evaporated down to 5° B., and then a strong solution of Glauber's salt added so long as a precipitate of sulphate of lime is formed. The whole of the acetate of lime is not, however, converted into sulphate of lime, accordingly the undecomposed remainder is precipitated as carbonate of lime by means of carbonate of soda. The liquid, freed from the precipitate by filtering, and now containing only sodium acetate and a little calcium sulphate in solution, is evaporated in shallow cast-iron pans to crystallisation; the resinous matters which separate during the evaporation are removed. The crystallising-point is reached at 15° B., the liquid is then drawn off into the crystallising tubs, in which, after four days, it deposits the sodium acetate in large crystals, which are now once or twice recrystallised.

The sodium acetate obtained is now fused in flat cast-iron pans. It first melts in its water of crystallisation, after the evaporation of which the salt again becomes solid; it must then be fused a second time at a higher temperature. The mass of salt then swells up, evolves oily vapours, and finally melts quietly; it is next removed from the pan, dissolved whilst still hot, and the solution filtered. The filtrate is left to crystallise, the sodium acetate decomposed by strong sulphuric acid in an iron pan lined with lead, with a copper or lead head, and the acetic acid distilled over; it is again rectified in a glass retort with a little pyrolusite. It must then be water-white, and free from foreign odour.

Preparation of Acetate of Iron from the Acetic Water of the Rosin Distillation.—The acid liquor obtained in the distillation of colophony is brought into barrels filled with small particles of iron—sheet-iron clippings, iron turnings and borings. In about eight to fourteen days the saturation of the acid is complete; it is accelerated by frequently drawing off the liquid, so that the iron in the barrels may come in contact with the atmosphere, which hastens the oxidation. The dilute acid in the barrels is also kept at a temperature of 25–31° C. by frequently injecting steam. However, the saturation is not always complete in the barrels; the solution is therefore drawn off, and heated to boiling in iron pans similarly filled with iron. The resinous matter which rises to the surface is skimmed off, and the process continued until the acid is completely saturated. The liquid is evaporated down to 10–15° B., and allowed to settle thoroughly before use. It forms a dark, blackish-green solution. Acetate of iron is extensively used in the cotton industry.

Utilisation of Rosin Oil and the Residue of the Distillation in the Manufacture of Soot-Blacks.—Rosin oil gives, when burnt in special stoves, a very fine soot, highly prized by manufacturers of lithographic and printing inks. In order to obtain the black from rosin oil, the latter is allowed to flow from a separate tank through a narrow pipe, whence it falls upon a red-hot plate contained in the stove; the oil is decomposed, the soot drawn into the depositing chambers, where it settles in various qualities. As soon as a certain quantity of oil has been burnt, and the stove, having stood for a sufficient time, is quite cold, the separate collecting chambers are opened, and the soot removed. The finest soot for lithographic purposes is always found in the last chamber, whilst a less fine black, used for letterpress inks, collects in the earlier chambers. The soot is graded by sieving.

The residue of the distillation, containing much rosin, must be burnt in a special soot-stove; it produces lower qualities of black, which are similar in value to the soot of ordinary flames. From the burnt residues the sodium carbonate may be regained by extracting, filtering, and evaporating.

CHAPTER XX

WASTE WOOD

THE utilisation of waste wood is worthy of attention for two reasons. In the first place, from a purely technical point of view, through the production of more or less valuable products from small and objectionable waste; in the second place, from the economic point of view, since nothing should be lost, and material once introduced in the manufacture must be worked up into new forms.

Perhaps, in a certain sense, necessity was the cause of the endeavours to utilise wood waste, as, for example, was also the case with blast-furnace slags, which, by their immense accumulations, diminished the working spaces of the works to such an extent that the removal of these by-products became a question of life and existence for blast-furnaces.

It is true that there is nothing to hinder the technical utilisation of waste wood, but, unfortunately, it is a hindrance that the waste is locally produced and must be utilised on the spot. Carriage of waste wood to any considerable distance renders its conversion into new products quite unprofitable.

In districts where waste wood is produced in large quantity and—what is very important—regularly, its utilisation is very profitable, especially when, by the development of special processes, products can be obtained which possess the natural good qualities of the wood, and also valuable qualities artificially added. On the ground of many years' experience, the author is strictly of the opinion that valuable imitations and complete substitutes can only be obtained by using the less valuable waste of the original substance. Imitations of, and substitutes for, leather can practically only be obtained by suitable treatment of waste leather; imitations of, and substitutes for, wood can only be produced from wood

waste. The industries which produce imitations and substitutes should abide by this principle; they would then be spared many disappointments, and we should read fewer patent specifications, of which it can at once be said that they are practically worthless.

Waste wood may be utilised in a variety of ways, but it would be unjust to designate all as equally good. Neither in execution nor in profit are they of equal value.

In regard to the appliances employed in utilising waste wood, it appears to be very difficult to give any accurate and particular statement, since the manner of working and the products obtained are too varied. Generally speaking, it will be sufficient to say that the necessary appliances are similar to those used in producing plastic masses,—mixing and kneading machines, moulds and presses.

If it be intended to consider seriously the manufacture of products from waste wood, it is first necessary to obtain clear ideas on certain points. The first question is always, as we have already remarked, Is there a certainty of a sufficient, reliable, and lasting supply of suitable waste wood? This question favourably answered, and the raw material thus secured in a sufficient and permanent supply, there comes the second question: Does the existing or main business possess any of the appliances or certain of the necessary conditions for the manufacture of certain products from waste wood? This question appears very important; it affects the question of profit. The last question to be raised is, Does the article which is to be made meet an actual need, and do time and circumstances appear so disposed that an abundant sale of the article is to be expected, so far as human estimates go, as the sum of the deliberations, comparisons, and experiences of the manufacturer and others?

These questions must first be drawn up, weighed, and answered. The more thoroughly and practically they are examined, and the more precisely the answers are drawn up, the more solid is the foundation for the manufacture and the better prospects has the intended process.

Special instructions for answering the first two questions cannot be given, because they are of a personal or local nature.

Hints may be given in regard to the answer to the last question, which will make possible a rapid and practical survey and a course of action.

The wood waste which is most important is *sawdust*, both on account of the quantity and frequence of its production. It is to-day utilised on a large scale. The most obvious and simplest method of use is as fuel, which has been before proposed, but was at first unsuccessful on account of the lack of suitable apparatus. Now, however, these difficulties have been completely removed by a rational construction of furnaces and grates, which enable fuels in powder or dust to be used without difficulty.

Another attempt, the first to touch on a real branch of manufacture, was to prepare **artificial wood from sawdust.** The binding medium used for the sawdust was glue solution and water-glass, also colophony, blood, and glue with potassium bichromate. The best binding medium for sawdust is glue, to which bichromate of potash is added in order to make the compound waterproof. On exposure to light, chrome glue, insoluble in water, is formed. The glue solution is prepared from five parts of good pale glue and one part of isinglass by softening, slow heating with water, and careful filtering. The quantity of water is decided by the nature of the glue; it ought not to be too small, but such that the liquid does not form a jelly on cooling, but only just begins to set. Moulds of metal, gypsum, or sulphur, after thoroughly oiling—or even of wood, if previously varnished with a spirit shellac varnish,—may be used for shaping the wood mass mixed with glue. A thin layer of the mass may be first applied, pressed well into the mould with the fingers, then the remaining space filled with mass prepared from coarser sawdust, the surface covered by a large plate, and weighted. Before taking out, which is easily done as soon as the mass is somewhat dry and coherent, the superfluous matter is cut off with a broad, thin knife, and the under surface of the relief thus made level. The articles may then be varnished or gilded, and treated just as ornaments cut out of wood.

The *wood paste* prepared by the method of Kletzinsky is

very well worth attention: 160 parts of sawdust—best from soft woods—are well boiled in a solution of 100 parts of sulphate of alumina in the necessary quantity of water, and then allowed to cool; 50 parts of glue are dissolved at the boiling-point in 100 parts of water. The glue solution is well mixed with the sawdust paste; the mixture is kneaded, rolled in press sheets, and pressed under a very powerful pressure. The pressed material, which is at first very brittle, on gradual drying in the air acquires a surprising degree of hardness. When sufficiently firm, it is moistened from three to five times with a dilute solution of potassium carbonate in water, after which it is finally dried. In this manner the separate particles of wood become united by a kind of alum-tanned glue, which is no longer soluble in water, and is of horny hardness. Any desired colouring matter may be added to the alumina mordant, and also ground dyewoods, in order to obtain coloured wood pastes. By adding coarse mixtures of wood meals of different colours beautiful mosaic plates for parquetry may be produced.

The property of sawdust to conduct heat badly has naturally been utilised. It has been recommended for lining ice safes, and is now used for this purpose. There is, however, no doubt that in this respect it leaves much to be desired, apart from the fact that a regular supply depends on local conditions, and that it is far from being the worst conductor of heat. (*Cf.* Infusorial Earth.)

Sawdust is used for *burning black clay pipes.* A layer of sawdust and a layer of pipes, 300–500 in number, according to size, are placed in a large muffle. The muffle is then luted up, brought into the furnace, which is built up, with the exception of the firing doors. The sawdust is carbonised by exposure to a red heat for 10-12 hours, and colours the pipes black. The pipes are next, in numbers of 20–50, placed on a round sieve with an equal number of holes, kept over a straw fire, when they become intensely black; they are finally polished by means of wax and a stiff brush.

Among the further processes in which wood waste is used is the **manufacture of oxalic acid.** In this case the operation is conducted on a large scale, and requires, besides

some chemico-technical knowledge, also suitable appliances, so that it appears advisable to introduce this application of sawdust only where a suitable manufacture already exists, to which that of oxalic acid might be added. In view of the many applications of oxalic acid and its salts, there is no doubt of the demand.

The process is commenced by preparing the lyes. Potash and soda are mixed in such quantities that, after causticising, the proportion of the potassium hydroxide to the sodium hydroxide may be as 40 : 60. The mixture of the two salts is dissolved in about eight times the quantity of water, and made caustic by boiling in an iron pan with slaked lime. After deposition of the calcium carbonate here produced, the lye is drawn off into another iron pan, in which it is evaporated to a specific gravity of 1·3–1·4. The sawdust, which is to be used, must be freed from large pieces of wood by sieving; it is then mixed with such a quantity of the caustic lye that there are two parts by weight of alkali to one part of sawdust. Since it is important that the whole mass of the wood should be uniformly saturated by the alkali, the concentration of the latter is arranged so that, after sufficient mixing, the whole of the liquid has been sucked up by the sawdust. The mass is then at once brought into the vessel in which the heating is to take place.

The yield of oxalic acid is the largest when the mass is spread out in thin layers; thus it is best to use for the heating very shallow dishes of about 2 metres (6 feet 6 inches) diameter, with rims about 5 cm. (2 inches) high, in which the mass is spread out to a depth of $1\frac{1}{2}$–2 cm. (0·6–0·8 inches). It is continuously turned over by a stirrer, which at each revolution lifts the mass from the dish, and allows it to fall back.

It is of the greatest importance that the temperature of the whole mass should be brought to 240° C., but should not exceed this limit. It therefore appears to be advisable to heat the pans, not by direct fire, but by the hot gases drawn off from the fire. This condition is attained by connecting a fire grate by flues with a low chamber, the roof of which is formed by the pans; in each flue is a damper, of careful

construction, which renders it possible to regulate the admission of the fire gases or to cut them off altogether. By using an arrangement of this description, it is not difficult to keep the temperature of the mass within the desired limits. The sawdust, saturated with lye, is brought into the pan by means of flat shovels, spread out, the stirrer put in motion, and at the same time the pans heated by opening the dampers. At first there is only the evaporation of water, the mass turns continually darker, until it becomes deep brown, and at the same time a peculiar smell begins to be evolved. When the temperature reaches about 180° C. the mass begins to turn pale again, and acquires a greenish-yellow colour. The temperature is now very gradually increased to 240° C., at which the mass is kept until particles of wood can no longer be seen in a sample withdrawn. The dampers are then closed, the melted mass removed with flat shovels and allowed to cool, or, what is better, at once treated further whilst still hot.

In the latter case the melt is thrown into a pan containing hot water; it rapidly dissolves, and heats the water almost to boiling. The heating is then continued until the concentration of the liquor is 38° B., when it is run out into small crystallising vessels, in which, during the rapid cooling, nearly all the sodium oxalate separates, so that only very little of this little soluble salt remains dissolved in the liquid, together with potassium carbonate, caustic soda and potash, and humus compounds. In order to free the crystals of sodium oxalate from the mother liquor, they are brought into a high tub with a false bottom, where the mother liquor drains off, and the remainder is then displaced by water. The mother liquors are evaporated to dryness, the residue freed from the humus compounds by roasting in the air, and the residual mixture of sodium and potassium carbonates again causticised.

In order that the soda united to the oxalic acid may be again immediately used in the form of caustic soda for the treatment of new quantities of wood, the sodium oxalate is treated in the following manner:—It is dissolved in a very little boiling water in a vessel heated by steam, and then thin milk of lime run into the hot solution, which is kept in constant motion by means of a stirrer, until the decomposition

is complete, *i.e.* until insoluble oxalate of lime and soluble caustic soda have been formed. The quantity of lime to be employed is calculated from the weight of sodium oxalate taken; since, however, both substances are not quite pure, the calculated quantity of lime does not agree with that really used. It is, however, important to bring no excess of lime into the precipitate, since otherwise in a later operation a larger quantity of sulphuric acid would be required than is needed to decompose the oxalate of lime. The precaution is therefore taken, after the greater part of the lime has been introduced, of taking repeated samples, which are filtered, acidified, and calcium chloride added; so long as a turbidity is produced, undecomposed sodium oxalate is still present.

The milky liquid is then run from the decomposing vessel into another, where it is allowed to settle, and the clear caustic soda solution drawn off from the precipitate. The latter is washed with water, and the first wash water, which still contains much caustic soda in solution, is added to the caustic solution; the further washings are, however, used to dissolve new quantities of the crude melt. The oxalate of lime, when sufficiently washed, is brought into a tank lined with lead, stirred to a paste with water, and then with the calculated quantity of sulphuric acid previously diluted down to 15–20° B. The quantity of the sulphuric acid is equivalent to that of the lime used in the decomposition; when two parts of sulphuric acid are used to one part of lime, the sulphuric acid preponderates, and there is obtained a solution of oxalic acid containing free sulphuric acid. However, it is advisable to work in this manner, since the excess of acid accelerates the decomposition of the oxalate of lime, and the sulphuric acid is utilised again in a later operation.

In order to hasten the decomposition of the oxalate of lime, steam is continuously introduced through a narrow lead pipe, which reaches to the bottom of the decomposing vessel. The solid compound is thus kept always suspended in the liquid; the heating also assists the decomposition. After the addition of the whole of the sulphuric acid, the liquid is allowed to rest, the solution of oxalic acid drawn off after the deposition of the gypsum, and the latter washed with water

in a lead funnel lined with linen, the first washings being added to the oxalic acid solution; the remainder are used in place of pure water to mix with the oxalate of lime in a fresh decomposition. The residual gypsum is an excellent fertiliser for clover, but may, since it is very pure, be dehydrated and utilised as plaster of Paris.

The solution of the oxalic acid is concentrated in a shallow lead pan, standing on a sand-bath, which is heated by an open fire, or better, by steam, until, in the summer, it is 15° B., in the winter, 10° B. It is then cooled to the ordinary temperature, when the dissolved gypsum separates in crystals. The liquid poured away from the gypsum is then further evaporated to 30° B., and set to crystallise in shallow lead vessels.

The mother liquors, separated from the crystals, contain the excess of sulphuric acid in addition to the oxalic acid; the sulphuric acid is estimated, and the liquors added in the next decomposition of oxalate of lime, the amount of sulphuric acid added being decreased by the corresponding quantity.

The oxalic acid crystals are washed with cold water to remove adherent mother liquor, and are then purified by solution in boiling water and rapid cooling, so that only small crystals may form. The compound is then sufficiently pure for commercial purposes; it contains a very small quantity of sulphuric acid, and an equally small quantity of oxalate of soda or potash.

In a certain sense **artificial vanillin** is a product obtained from waste matters. In the year 1861, T. Hartig found a crystalline substance in the cambial sap of the coniferæ, which he termed "laricin," but later, after he had ascertained its presence in the majority of coniferous trees, "coniferin." It is obtained in the following manner:—Pines, firs, larches, felled between the middle of May and the middle of July, are gradually stripped of their bark and bast layers; the young wood fibres, swollen with sap, are scraped off by means of pieces of glass from the hard woody layers, and collected in vessels beneath. The scraped-off material is at once separated from the sap by fine press cloths, and the sap immediately boiled. The coagulated albumin and the

solid substances it encloses (cell nuclei, starch grains) remain behind on filtering through paper, whilst a clear filtrate is obtained, which, when carefully evaporated to about one-fifth of the original volume, separates the cambial sugar, and also the coniferin in crystalline form. By treatment with cold water, the difficultly soluble coniferin is separated from the sugar, it then crystallises in white needles, generally united to rosettes, which give a deep violet coloration with strong sulphuric acid.

Dr. Haarmann, of Holzminden, has given a similar account of the preparation of artificial vanillin in Post's *Zeits. für d. chem. Grossgew.* The raw material for the preparation of artificial vanillin, says Haarmann, is coniferin, a glucoside contained in the cambial sap of coniferous trees. It is obtained in the districts where the trees are felled during the sap period—Thüringia, the Baden and Würtemburg Black Forest. The coniferæ—red and white firs—are barked, the sap lying below the bark, together with part of the bast, scraped off by means of a piece of glass or a knife, and collected in a vessel. The sap is filtered from the bast as rapidly as possible and boiled, since it readily ferments, and then completely decomposes. The liquid is then separated from the coagulated albumin, evaporated down to one-fifth its volume, and set aside to crystallise. After a short time the crystals produced are pressed to remove the mother liquor; 100 litres of the sap, which is very difficult to obtain, produce 0·5–1 kilo. of coniferin. The coniferin is then treated with oxidising agents (sulphuric acid and potassium bichromate), and the vanillin either distilled in steam or extracted by ether. In order to separate the vanillic acid produced, the substance is subjected to purification by sodium bisulphite solution and then recrystallised. After this operation, vanillin forms an almost white crystalline powder, which melts at 80–81° C. Twenty grms. of vanillin are equivalent to 1 kilo. of the best vanilla, since the latter contains an average of 2 per cent. of this compound.

The manufacture of pyroligneous acid from sawdust may be applied in all cases where the production of acetic acid from alcohol is not profitable. One hundred kilos. of sawdust

give 45–54 litres of a liquid, which contains 4 per cent. of acetic acid, and also 6–8 litres of tar.

The dry distillation of sawdust begins at a temperature of 100–130° C., and at first only water comes over, the quantity of which naturally depends on the more or less moist condition of the sawdust. According as it has been kept in the open or in closed rooms, it will contain a larger or smaller quantity of moisture, which influences the quantity of water distilling over, and also the consumption of fuel, which materially depends on the quantity of water. It is therefore advisable, when possible, to give the sawdust a preliminary drying, and then, when well dried, to bring it into the distilling apparatus. The temperature rises from 145–500° C.; the products of distillation are water, pyroligneous acid, wood spirit and tar, also various gaseous substances, whilst charcoal remains. The acid products form the chief quantity when the temperature is rapidly raised; they must be rapidly removed if they are not to undergo a further decomposition.

The *application of sawdust as an addition to cement*, in order to prevent the formation of small cracks, is technically interesting. The sawdust is well dried, then sieved in an ordinary sieve so that only the small woolly flocks are used. The mortar is mixed from one part of cement, two parts of lime, two parts of sawdust, and three parts of sharp sand. The sawdust is first mixed dry with the cement and sand, and the lime then added.

Wood waste finds another application in the **manufacture of casks.** The outer skin of the stems, removed when the tree is sawn up, is cut up by an ordinary circular saw into lengths equal to the length of the staves required. These lengths are now cut up into proper widths by means of a circular saw with two to three blades, which can rapidly and easily be put in motion. The wood is fed to the saw by means of rollers with deep grooves and running in elastic bearings. The pieces now come to a circular saw with a vertical spindle and horizontal roller feed, by which they are cut to shape. The saw blade has a diameter of 23–24 inches. In the middle of the spindle above the table is a guide, to right

and left of which, in front of the saw, and thus diagonally opposite, are grooved rollers in elastic bearings. By means of this arrangement two pieces can simultaneously be cut to different thicknesses. The staves now go to a trimming and slitting machine, in which they are cut into exact lengths and bevelled towards the inside, and the slits cut for the reception of the ends. This machine has a shaft upon which the slit-cutting tool runs, and two other shafts upon which run the circular saws which trim the staves. The saws and tools can be fixed at different distances apart to allow for varying lengths of the staves.

The staves are fed in by hand, each being laid with its hinder end against a guide, which can be moved backwards and forwards. The pieces of wood after this treatment, come to the jointing machine, in which the staves are carried in a segment-shaped guide over a slit below which is a small stout circular saw. The staves receive in this machine an accurate joint, which makes subsequent working by hand unnecessary, and enables the body of the cask to be at once built up. For the sake of easier carriage, which is important, the casks are tied up in a bundle. The ends of the casks are cut from smaller waste pieces in a similar manner to the staves. For this purpose wood is used which, by its structure, is unsuitable for staves. The separate pieces are pegged together, and finished on a machine which cuts out the round end. All the machinery may be tended by youths. With one set of machines several hundred casks can readily be made in a day. The sale of these casks, made from outer skins of trees and waste pieces, should be almost without limit; cement makers, millers, nail makers, and fruit dealers in particular buy them readily.

The application of the **waste of saw-works in manufacturing parquetry** must not be overlooked. Parquetry sheets are made in sizes of 59 cm. and 64 cm. square (24 and 26 inches square). The foundations for inlaid parquetry are made by gluing together strips of wood somewhat shorter than the dimensions of the sheet, cutting these glued pieces into the right length, grooving at the ends, and finally grooving and tonguing. In Russia, another

method is adopted; the foundations are made from a frame with two middle pieces and four panels. The frames are so morticed and tenoned that each piece has a mortice at one end and a tenon at the other. The four panels are grooved and tongued into the frames and the cross pieces, only the tenons being glued of the whole work. The panels are inserted so that the grain of the wood may run in one at right angles to the direction in the other. By this arrangement warping is made impossible and shrinkage is reduced to a minimum, since only the two parallel frame pieces in a total width of at most 25 cm. (10 inches), under the most unfavourable conditions of temperature, can swell or dry to a small extent. All wood waste may be used in making these foundations, since the panels have at most a length of 20 cm. (8 inches). These parquetry foundations can easily be made by means of a few circular saws and a very simple boring machine for making the holes.

Sawdust for the Production of Stoppers.—F. Koeller, of Neulengbach, in Lower Austria, has obtained a patent for a method of making stoppers from sawdust. The sawdust is rolled round a round wooden rod; the sawdust is made to adhere to the surface of the rod by means of some binding medium such as rosin or indiarubber cement. The rod has the same length as the layer of sawdust; it furnishes a safe hold for the corkscrew in removing the stopper from the neck of the bottle. Finally the stoppers are dipped to half their depth in pure melted paraffin, and are then ready for use.

Finally the **manufacture of briquettes from sawdust** is to be mentioned. Molasses is recommended as an excellent binding medium for the production of briquettes from sawdust. The latter is moistened with the dilute molasses, the whole intimately mixed, then brought into cylindrical, brick, or other shape by means of suitable presses, and dried. Colophony and tar may also be mentioned as binding agents. The prospects for the success of the manufacture of briquettes naturally depend, in the first place, on local conditions, especially on the regular supply of the waste material.

CHAPTER XXI

WASTE HORN

Utilisation of Turnings and Filings.—The waste materials are mixed with a saturated solution of potash and lime, by which the horn substance is attacked and finally transformed into the condition of a jelly, in which state, by the application of a gentle heat, it may be cast into moulds and pressed, when the mass loses moisture and becomes hard and coherent. A final pressing, under the influence of heat, gives the moulded mass its final form; it may then be worked up into the most varied articles—pipes, cigarette-holders, stick handles, buttons, etc.

Horn waste may also be utilised in the following manner: —The turnings, raspings, and filings are moistened and pressed into a cylindrical metal mould by a metal piston to a solid cake, heat being applied. The mass is then rasped to a fine powder, which is again pressed in the same manner; the operation is repeated until the mass has acquired a sufficient degree of hardness and solidity. Finally, it is again rasped and carefully sieved, so that all the coarser particles are removed. As a rule, horn and tortoise-shell waste are worked up together; the articles are then less brittle than when made from tortoise-shell alone. From the fine powder the finished substance is obtained as follows:—The powder is brought in layers between brass plates; several such layers are placed under the press, and this brought into boiling water, when the mass becomes hard and coherent. The plates are then further worked. Finished objects may also be pressed out of this mass, if the necessary moulds are provided.

Horn filings may be brought by the following process into such a condition that, from the mass, stick and umbrella

handles and many other articles may be made by casting in moulds. One kilo. of quicklime, 500 grms. of potash, 40 grms. of tartar, and 30 grms. of common salt are dissolved in water, and then one-third of the quantity of water evaporated off. The rasped horn or turnings is then thrown in and boiled, until the mass becomes so thick that it can be poured into a mould, which must be well oiled, whether it be of metal, wood, or earthenware. If it is desired to colour the horn, the necessary colour is stirred into the fluid mass before casting.

The waste horn may also be boiled in a strong lye of potash and lime, until the mixture is thick enough to be poured out and moulded; before casting, a colouring addition may be given.

In order to obtain solid horn from waste, Pathe lays it for an hour in a liquid consisting of a cold saturated solution of boric acid in water, and a cold saturated solution of arsenious acid in dilute hydrochloric acid; the quantity of boric acid should be twice that of the arsenious acid. The vessel containing the horn substance, swollen by this treatment, is then placed for an hour in a water-bath at about 60° C. Finally, the horn substance is subjected to considerable pressure by means of a piston in a closed iron mould, heated to 120° C., until all the liquid is removed. After cooling, the pressed mass forms solid plates of horn, which can be worked like natural horn, and are characterised by pliability and elasticity.

CHAPTER XXII

INFUSORIAL EARTH

Utilisation in the Manufacture of Ultramarine.—The application in the manufacture of ultramarine was the first introduction of infusorial earth in industry. In making ultramarine rich in silica, infusorial earth was added to pure clay, in place of quartz sand. It is certain that it has been much used for this purpose in recent times. Infusorial earth is a very *bad conductor* of heat; it is accordingly used for filling the insulating spaces of ice-cellars; similarly it is an excelling filling material for safes and ice-boxes. It is especially to be recommended for *cleaning glass plates* for photographic purposes. Even very greasy glass plates rapidly become clean when rubbed with infusorial earth moistened with water. Also, when it is required to use acids, alkalis, and solvents for cleaning purposes, they may, in a certain sense, be used in the solid form, when mixed with infusorial earth, which is able to take up more than double its weight of liquid without running. Infusorial earth is a very valuable material for *glass-making*, since it accelerates the formation of the glass by readily mixing, on account of its fine state of division, with the other materials. Similarly, infusorial earth is an excellent *grinding material* for hollow glassware.

In preparing water-glass, 74·5 parts of calcined soda are dissolved in five times the quantity of boiling water, 42·5 parts of slaked lime added, and the mixture kept at the boil until a small portion of the liquid, when mixed with an acid, no longer effervesces. After the deposition of the chalk, the clear liquid is syphoned off, the chalk again boiled with water, the two liquids united and evaporated down to a specific gravity of 1·15. To the boiling liquid 120 parts of finely powdered infusorial earth are gradually added; after

this has dissolved, 3 litres of lime water are added. There is then produced a brown precipitate, which removes from the solution the organic matter contained in the infusorial earth. The liquid poured off from the precipitate, after it has been evaporated to a syrupy consistency, solidifies on cooling to a yellowish jelly, which dries completely in the air, and readily dissolves in hot water. 120 parts of infusorial earth produce 240–250 parts of a jelly, which contains 47 parts of anhydrous soda water-glass and 53 parts of water.

Infusorial earth finds a further application in the **manufacture of light bricks**; 24 parts of Lüneberg infusorial earth, with 1 part of fat clay, give a mass which works well. The infusorial earth, which has generally too little binding power, requires an addition of binding clay, with which the very fine earth can be mixed only by a very careful process of mixing up together with water; also the temperature of the porcelain kiln, which is rarely reached in a brick kiln, is required for burning.

CHAPTER XXIII

IRIDIUM FROM GOLDSMITHS' SWEEPINGS

D'HENNIN removes the iridium from goldsmiths' sweepings containing that metal (according to *Dingler's Journ.*) by fusing 12·5 parts with 3 parts of sodium arsenate, 18 parts of black flux, and 20 parts of ordinary flux—borax, tartar, charcoal, and litharge—and separating the lead regulus containing the gold and silver from the upper layers of iron-grey lumps consisting of iron, iridium, and arsenic. In France (according to Muspratt) large quantities of this refuse are worked up; they come from America, principally from Californian gold, which often contains osmiridium. By repeatedly melting gold alloyed with copper, according to Wilson, the osmiridium separates almost completely. According to Dubois, the Californian gold is melted with three parts of silver, when the osmiridium deposits from the alloy, which has a specific gravity of 12–13.

CHAPTER XXIV

JUTE WASTE

Treatment.—The softening, beating, and carding machines are used in treating jute waste (E. Pfuhl, *Dingler's Journ.*, 222, p. 573).

1. *Jute Cord and Fabric.*—Both are collected and go into the store as the first waste product. The latter is used for packing purposes. The former is first unknotted by hand-labour outside the factory, then untwisted or unravelled, and cut into lengths of about a yard, which are laid parallel to one another, made up into bundles of about 45 lb., and tied together. A large number of these bundles go into the softening house, where they are sprinkled with water and fish oil, allowed to lie in layers, and then taken to the breaker card. The jute cord is treated either alone or mixed with other jute.

2. *The root ends,* which have been cut off, are worked up in the same manner as the cord into the lowest yarn numbers, and are spread out on the feeding-table of the corresponding breaker card. Other waste, which is formed at times, is, instead, taken to the finisher card, which treats the corresponding qualities. In order that markedly different slivers may not be produced by too rapid supply, these tow slivers must be introduced slowly and as thin as possible over the feeding rollers. The moment of passing the rollers is the best for adding faulty slivers.

3. (*a*) *Carding Tow.*—In the first place, the shorter strips of fibre are sorted out by hand, and again subjected to the carding operation, after which they are given a beating or shaking treatment. The lower qualities of this tow are separately collected and stored in suitable places in the open; they are removed by cartloads for manure. The better qualities are compressed in box-presses to bales weighing

2–4 cwt., which are tied up and sold to paper or pasteboard makers.

(*b*) *Spinning Tow* is not subjected to a special treatment; either alone or mixed in the shaking machine with the breaker tow, it is pressed into balés, packed, and sold to papermakers.

(*c*) *Roving Tow* can be readily introduced into the spinning process. It is either placed on the cloth of the breaker card together with long jute, or, better, is passed through the softening machine, and then taken to the finisher card for further treatment, for which purpose this machine must be supplied with a feed-table; the fibres then remain longer. The roving tow should, however, never be added for re-treatment to medium or better qualities.

(*d*) *Good Tow from the Fine-spinning.*—The dropped threads of the rovings are sorted, mixed with the roving tow, and spun together with it. A second sorting of the tow then takes place in the softening house, after which it is sent once through the softening machine and then over the tow-carder, when a loose and very soft cleaning material is obtained, which is packed in sacks, or, better, compressed into bales, and sold to railway workshops, etc. This tow is not suitable for spinning.

(*e*) *Clean Thread Waste.*—This is sent once or twice through the softening machine, when it also gives a very fine clean cleaning material, which, however, is somewhat less soft than the preceding substance. The waste, after this treatment, is often used for stuffing ordinary mattresses, sofas, etc., in place of flax tow, in comparison with which it has the advantages of cleanness and purity, but has, however, the smell of fish oil.

(*f*) *Ordinary Sweepings.*—After sweeping together, any good waste, such as sliver, roving tow, etc., is sorted out; then follows the search for oily cleaning waste, which, since the presence of oil is regarded as producing danger of fire, is sent to light the fires in the boilerhouse, than which no better utilisation can be found. The remainder is either at once mixed with the beaten ordinary carding-tow, or is previously beaten, when any better fibres remaining in the machine are united with the tow from the breaker card.

The tow-cleaning machines may be divided, according to the nature of the beating apparatus, into—(*a*) simple beating or shaking machines, (*b*) conical shaking machines, and (*c*) double shaking machines. Of these three machines, the most practical are the first and last.

Simple Beating or Shaking Machine.—In a cylindrical vessel, the upper half of which is a tight casing with a door for admitting and removing the material, and the lower half a grating of laths, moves a horizontal shaft provided with fast and loose pulleys. On the shaft are fixed six series of round iron rods as beaters. As the shaft rotates, the beaters move between two other series of fixed rods. The side walls of the machine are well boarded, so that the dust-chamber is formed under the grating. After the waste (carding tow) has been introduced through the above-mentioned door, and distributed throughout the machine, the door is closed, the shaft set in motion, and the beating continued for ten to fifteen minutes. The shaft with the beaters is then removed, and the machine emptied of the cleansed waste through the door. The dust, sand, bast particles, etc., beaten out, fall through the grate into the dust-chamber. They are most readily and conveniently removed from the workroom if the machine is erected over a brickwork pit, connected by means of a passage with a special dust-shed in the open. If the drainage conditions do not permit such a pit and connection with a shed, the shaking machine must be erected at a greater elevation, and a large box running on wheels brought under the dust-chamber, in which the ordinary refuse shaken out can collect. If the dust-chamber is connected with a fan which carries away the lighter dust to deposit it in a suitable position, it is still necessary to catch the heavy particles separately. The beater-shaft generally makes 260–280 revolutions per minute.

Double Beating Machine.—Two horizontal shafts, each with six series of iron beaters, are so arranged in a cylindrical casing that the rods of one move through the spaces between the rods of the other. The casing also here consists of an upper tight cover and a grating below. The beaters in addition strike through another series of rods. The charging

opening is closed by a counterpoised door during use. The dust-chamber communicates either with a pit or with a closed box; a fan may also be used. The feed is fixed in the upper part of the cover; it consists of an endless cloth, a small loose roller, and a roller, studded with needles, moving at a high speed,—both in a tight-fitting casing. The needle roller is designed to introduce the material, as far as possible, into the beater in an unravelled and divided state. A certain quantity of waste is brought into the machine, and the feed set in motion. In a few minutes the door is opened, so that the cleaned material might be thrown out and caught in a box placed for it. The door is then closed, and a fresh quantity introduced. This repeated change can readily be worked automatically by suitable mechanism.

The Tow Carding Machine or Teaser Card.—A rotating drum, to which the material is brought by the feeding apron and grooved rollers, is surrounded on the upper half by three pairs of turning and squeezing rollers working together. These rollers treat the material in the known manner; it then goes to the delivery roller, from which it is drawn off by smooth rollers as a coherent fleece and carried on to the delivery apron. The machine is therefore a semi-circular card with an upper working half. In order to prevent the production of dust, the rollers are all protected by a sheet-iron cover. The diameter of the drum is generally 3–4 feet; it is about the same length, and runs at 100–120 revolutions per minute. The rollers move at various velocities, which have to be adjusted to the needs of the moment. The ratio of the velocity of feed to that of delivery should be at the most 1 : 15.

CHAPTER XXV

CORK WASTE

Utilisation of the Waste of Cork-Cutting.—The waste is either at once used for stuffing mattresses, for upholstery, or ships' fenders; or it is sorted, the adhering bark removed and applied to the same purposes; or it is brought into powders of varying degrees of fineness (the chief use). The waste is first, by hand labour—at a low price—and sharp knives, freed from the outer rough and dark layer of the bark, then cut into sheets of different thicknesses, and finally into cubes of different dimensions.

The waste is also treated by filing or rasping, by which means a more or less fine dust is produced. It is easily comprehensible that the increasing fineness of the cork dust considerably increases its price.

The woody portions of the waste, as well as the peculiar hard, sandy powder found in the cork, are partly separated by picking, partly by sieving, so that the remainder is, as far as possible, free from these impurities. Special machines are used for grinding cork waste, among which is to be mentioned the patented "Favorita" mill, of H. R. Gläser, of Berlin. This machine consists of a cast-iron base, upon which is the real mill, with the grinding arrangements, the feeding arrangement, the transmitting disc, and the regulator. The mill works by reason of two conical surfaces, each provided with teeth of triangular section concentrically arranged, and engaging together. One of the cones rotates, whilst the other is stationary and fastened to the framework. The grinding teeth pass by one another in such a manner that the material, introduced at the apex of the cone and driven by centrifugal force towards the base, is cut as by shears, and crushed. The grinding surfaces may be moved nearer

together or farther apart by a hand wheel whilst the mill is working, so that the cork may be ground to different degrees of fineness. The teeth are fixed in segment-like plates, which are arranged for renewal; and according as the teeth on these segments are smaller or larger, near together or wide apart, the mill delivers finer or coarser cork. The velocity of the rotating cone also affects the fineness of the product.

Utilisation of Cork Waste for Gas-Making.—Illuminating gas may be made in the usual manner, in closed retorts, from cork waste, 100 kilos. (2 cwt.) of which give 50 cubic metres (1850 cub. feet) of gas. The distillation is naturally much more rapid than that of coal. The illuminating power of the gas, with a consumption of 150 litres (5¼ cub. feet) per hour in the bat's-wing burner No. 10, is thirty-six candles, so that there would be a saving of 50 per cent., in comparison with coal-gas at the same price, in view of the greater illuminating power. The cork tar produced as a by-product is of the consistency of ordinary tar, and reddish-brown in colour: on fractional distillation it gives 27 per cent. of light oil passing over at 210° C., which consists mainly of benzol and toluol with a little naphthalene. The heavy oil contains anthracene and a little phenol. The liquid condensed in the gas manufacture contains methyl alcohol, acetic acid, and ammonia.

Cork Waste for Cork Mattresses.—In making these mattresses a fabric as waterproof as possible is sewed or glued together so that it forms a large sack, which is then filled with cork waste or coarse ground cork, sewn up, and then given the shape of a mattress by quilting. In order to make it quite impenetrable by water, the seams, and especially the quilted portions, are coated with an indiarubber solution, after the drying of which the mattress may be used. Mattresses for gymnasia are made in the same manner, but using an ordinary coarse fabric. Ships' fenders are bags or baskets filled with cork waste.

Use of Cork Waste in the Vinegar Manufacture.—It has been recommended to replace wood shavings in the manufacture of vinegar by the much lighter cork waste. The elasticity of cork is made still greater by wetting, so

that a sinking together of the filling material is not to be feared even in high vinegar vessels. In the pores of cork are countless small organisms, and among them the acetifying bacteria in great quantity, so that the vinegar-making vessels filled with cork rapidly turn acid.

CHAPTER XXVI

LEATHER WASTE

LEATHER cuttings are made into artificial leather, which can be used for the welts and heels of boots. The manufacture is very simple (according to the *Deutsche Ind.-Zeit.*). The leather waste is made, with the addition of a binding medium, into rectangular plates, which are put one on the other, pressed in a hydraulic press, dried, and rolled. It is at once evident that this leather is only suitable for cheap work, and cannot be used where it is exposed to the action of water.

The process of Sören Sörensen, of Copenhagen, is as follows:—If the leather waste is impure, it is first freed from all foreign constituents, after which it is converted into a uniform fibrous material in a machine constructed for the purpose. When this finely divided leather is mixed with ammonia solution, a gelatinous mass is formed, which, when pressed into moulds or rolled into sheets and dried, gives a very hard and stiff material of considerable cohesion, but without elasticity, and soluble in water. In order to make it elastic and resist the action of water, it is mixed with indiarubber. The rubber, of which the finest Para to the commonest African qualities can be used, is squeezed and washed in the washing-machine, which consists of two grooved steel rollers, over which is led a stream of water, which has the double effect of washing the rubber clean and preventing it from being burnt by the great friction. After washing, the rubber is dried, cut up, and dissolved by means of spirits of turpentine, benzine, carbon bisulphide, or other suitable solvent. The quantity of rubber to be dissolved varies according to its quality. Para rubber is dissolved, or, rather, gradually swollen, by 4 parts of solvent; Central American scraps require 3·5 parts, Guayaquil rubber 3 parts, and African rubber 2·5 parts. The

rubber so prepared is then mixed with the ammonia solution, and the mixture well stirred. The proportions of the mixture depend on the quality of the product to be made. For example, for soles, 25 parts of solid rubber, 67 parts of ammonia, and 67 parts of leather; for heels, 25 parts of rubber, 80 parts of ammonia, and 80 parts of leather; for welts, 25 parts of rubber, 75 parts of ammonia, and 90 parts of leather. After kneading until the mass is quite homogeneous, it is dried. During the drying it is subjected to progressive pressings, the intensity of which varies according to the destined application of the product. For sole leather the greatest pressure is used—about 450 kilos. per sq. cm. (about 2¾ tons per sq. in.). After pressing, the substance is either painted or lacquered, or treated in some other way to give it a similar appearance to natural leather.

Smith and Johnson, of Huntington, dissolve the waste leather in sulphuric acid, pyroligneous acid and fusel oil, then add melted wax, and mix the whole intimately with paper pulp, which has also been previously impregnated with wax. The mixture is then dried, ground, steamed, and pressed in the desired moulds. Such leather is said to be waterproof, and very durable.

The leather made by the following process, published in Ackermann's *Gew.-Zeit.*, is as pliable and durable as ordinary leather of the same thickness, may be used for the same purposes, and is equally waterproof. The mixture consists of 1 lb. of indiarubber to 3¼ lb. of leather waste of any kind in the form of raspings. In order to mix these substances intimately, the rubber is dissolved in benzine or carbon bisulphide; when dissolved, 1 lb. of ammonia is added and the mixture well stirred. The rubber is then precipitated with a greyish-white colour; the leather waste is next gradually kneaded into this pasty mass. When the mixture is homogeneous, the stiff paste may be rolled or pressed into sheets, cords, pipes, rings for making cold-water pumps tight, flanges, and valves. The product is better than vulcanised rubber or leather alone.

Leather waste may also be used in the **manufacture of artificial inlaid ivory,** which is said to be obtained from the

bones of goats and sheep and pale or buckskin leather. The bones are treated with bleaching powder for ten to fourteen days, then washed in clean water and dried. When this is done, the bones and the leather waste are dissolved together in a pan with steam, so that a uniform fluid mass is obtained. To 10 lb. of this mass $\frac{1}{4}$ lb. of alum is added, and when this is well mixed, the scum which has risen to the surface is skimmed off, until the mass is quite clear and pure, then whilst still lukewarm the necessary colours are added, the mixture strained through a clean linen cloth, and poured into the requisite moulds. When sufficiently cooled the cast mass is removed from the moulds and laid on linen stretched over a frame, upon which it dries in the air. When quite dry it is soaked in a cold solution of alum for eight to ten hours, until it has attained the required hardness; 1 lb. of alum is used to 2 lb. of inlay for this purpose. When taken out of the alum solution the inlay is washed with fresh water, and again dried on the frame mentioned before.

H. P. D. Lissagaray has obtained a patent for a new process for manufacturing an assimilable **fertiliser from leather waste.** The waste is immersed for about five minutes in water to which has been added about 10 per cent. of strong sulphuric acid, or a sulphate such as alum or manganese sulphate. It is then dried in the open air, and finally in a current of hot gases. The material has now become quite friable; it is ground in a suitable mill to a fine flour, which is readily accomplished.

Leather Waste for the Manufacture of Glue.—In regard to the application of waste leather in making glue, J. Repp has constructed a machine for disintegrating the leather. It consists of a cast-iron box 3 metres (9 ft. 10 in.) long and 1 metre wide (3 ft. 3 in.), with rounded angles. The box is divided by a sheet-iron partition 5 mm. thick ($\frac{1}{5}$ in.) into two parts, which are connected at the round sides by the water which circulates through. At one end the water enters under the supply pressure, it passes through one division, and then back through the other to the outflow. The box is made in two pieces, at the top is a horizontal shaft with a roller studded with knives. The middle of the shaft is 640 mm.

(2 ft.) from the bottom, the roller is 650 mm. (2 ft. 2 in.) in diameter; the height of the box is 1000 mm. (3 ft. 3 in); it is provided with strengthening ribs. The knife cylinder consists of a drum in which is screwed a knife projecting 42 mm. ($1\frac{5}{8}$ in.); below it is brought a long knife, which can be adjusted at greater or less heights, according to the treatment required. The roller is driven by a belt at a speed of 120 revolutions per minute, thus the water and the leather which is being washed in it circulate through the roller, where the leather is cut to pieces. A charge of leather is 200, 150, or 120 kilos. (4 cwt., 3 cwt., or $2\frac{1}{8}$ cwt.), according to the size and speed of the apparatus. In setting the machine in motion the box is almost filled with water, for which purpose it is connected with a pump or water supply. The working side, in which are the roller and the arrangements beneath, is somewhat wider, thus the leather stands at a rather higher level on the other side, in consequence of which it approaches with a certain velocity, due to the difference in level. The throat consists of a cast-iron frame with a wooden lining, upon which the long knives are fixed and screwed. This throat at its upper concave part occupies almost one-quarter of the circumference of the roller, with which it is concentric, it falls away at both sides in an inclined surface of iron or wood. The cooled leather is well divided in this simple apparatus, which requires little attention; at the same time it is washed clean, so that it may at once be used in glue-making. The apparatus works excellently, since the leather is better purified and divided by the constant flow of clean water than by the older methods, in which the leather was first hung in the water in large baskets on a chain and then was ground after again drying. This apparatus is termed a hollander, but is not to be confused with the hollanders of paper-works.

Leather, which has been tanned with a substance insoluble in water, such as chamois or bark-tanned leather, is not at once suitable for glue-making; it requires a complicated treatment, which, however, is always profitable. In the case of bark-tanned leather, the manufacturer has to make sure that all the tannic acid is removed from the animal tissue,

since only a small quantity of this substance is sufficient to remove from the tissue its property of dissolving in water, and thus of producing glue.

In the first place, it is important that the leather waste should be converted into the finest state of division, for which a half-stuff hollander, such as is used in pasteboard and paper-works, is the most suitable (F. Dawidowsky, *Die Leim- und Gelatine-Fabrikation*, Vienna). The advantage is that the leather is not only disintegrated and washed in a manner which renders it very suitable for glue-making, but that it can also be used in the manufacture of leather book-covers, which are very soft and of good appearance when made from leather stuff mixed with rag or wood pulp.

When the leather waste has been so prepared, and, in particular, well washed, then a chemical treatment follows. Stenhouse uses a boiler at two atmospheres pressure, and 15 per cent. of lime to the leather mass to be treated, the lime being suspended in much water. Others use, to extract the tannin, caustic soda of 1·025 specific gravity, with which the leather paste is boiled for twelve hours. After boiling, the water is run off, the paste pressed, and again boiled with caustic soda of the same strength. The soda is now carefully washed out, which is done in the hollander. If the caustic soda in the liquor first drawn off is neutralised, the solution can be used for tanning or as tannic acid for other purposes.

The following is another process:—1·5 lb. of oxalic acid is dissolved in 12 litres (21 pints) of water, the boiling solution poured over 50 kilos. (1 cwt.) of leather waste, and the mixture kept in the water-bath at a temperature of 80–100° C. The leather should then dissolve; when this is the case, 15 litres (26 pints) of water are gradually added until a uniform mass results. The paste obtained by slaking 5 lb. of quicklime is then added, and the whole well mixed, when the mass becomes friable and powdery. It is now rubbed through a wire sieve, and the still moist mass exposed to the air. After three to four weeks the tannic acid is destroyed, and the mass becomes lighter. The lime is removed by means of water and hydrochloric acid. If all the tannic acid should not be destroyed by the exposure to air, in boiling

out the crude glue, 0·5 kilo. (1·1 lb.) of ammonia and 0·5 kilo. of ground pyrolusite may be added to 50 kilos. (1 cwt.) of the leather. Frequent turning over and gentle heat during the exposure to air hasten the process of decomposing the tannic acid.

The fatty ("stuffed") waste of the tanner and currier is utilised, according to J. Bergmann (*Neueste Erfindungen und Erfahrungen*, 1894), by the following process:—One end of an ordinary barrel, best of hardwood, is removed, the barrel placed with the open end upwards, and a perforated false bottom inserted at a height of 15 cm. (6 in.) above the bottom. A steam-pipe of 13–20 mm. bore (0·5–0·8 in.), according to the size of the barrel, passes down in the barrel to the perforated bottom; a valve for regulating the supply of steam is placed above the barrel. The waste to be extracted is filled into the barrel to one-third its height; the valve on the steam-pipe is opened and steam sent in, until the leather, which is stirred two or three times during the fifteen minutes' passage of the steam, is heated, which may be recognised by its dark brown colour. During the boiling, the fatty matter contained in the leather becomes free, and appears on the surface of the separate pieces, whilst the condensed water collects between the two bottoms, whence it may be run off as necessary by an outflow tap.

After this treatment the leather is at once shovelled out of the barrel and put in layers of 3–4 cm. (1·2–1·6 in.) into press cloths of equal size. The press cloths containing the leather are now laid in piles in a vertical spindle press, the plates of which have been warmed, in such a manner that upon each layer of waste comes a metal plate, and that there may be ten or more such layers, according to the press, above one another. As the press-plate attached to the spindle descends, the fat is pressed out of the leather waste; it flows away until it becomes thick by cooling, which may be prevented by arranging under the bottom plate of the press a double bottom permanently heated by steam. As soon as the fat ceases to flow away, the spindle is screwed back, the cakes removed from the press, the cloths removed, and the naked cakes set out to dry. The fat obtained by pressing, which is absolutely

pure, and has not in any way suffered by the action of chemicals, may at once again be used for stuffing hides and skins. As a matter of fact, it is better than the fresh fat prepared from tallow, dégras, and cod oil, since any resinous constituents or other injurious substances have been removed by the above process. The dried press-cakes are then ground, producing an excellent fertiliser, which can be used without any other addition, and finds a ready sale in the market.

R. Jäkel, of Berlin, has patented **a machine for producing so-called artificial leather.** In Fig. 9, *A* and *B* are two smooth drums; *C*, *D*, and *E*, three smooth massive rollers. The bearings of the drum *B*, and of the rollers *C*, *D*, and *E*, can be fixed by screws in any position. *A* and *B*, also *C* and *D*, rotate in their bearings, whilst *E* is fixed. The pattern is stretched round *A*, *B*, *C*, *E*, and the ends stitched together.

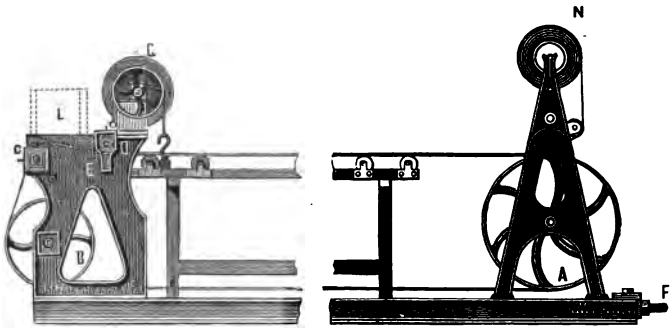

Fig. 9.

The machine is driven from the drum *A*, which moves both the pattern and the system of rollers.

G is a roller, upon which is wound the backing; its spindle rests at one side in an open bearing, and at the other in a hole, so that it can readily be changed. A brake acting on the projecting spindle keeps the fabric stretched, and ensures a regular feed. *L* is a sheet-iron pan with double walls, kept full of hot water in order to keep the mass in the pan hot; its width is equal to that of the machine. The mass flows out of the pan *L* on to the pattern, where it is distributed by a movable guide towards the sides, and is then spread on

in the desired thickness by means of the rollers *D* and *E*, or, rather, by passing between *D*, which is covered by the backing, and the pattern; at the same time the mass receives the design from the pattern. The mass cools on its way to the drum *A*, when it is finished; it is then wound on the roller *N*, which is turned by a handle, *O*.

In order to obtain the negative, or pattern, of a material such as leather, damask, etc., the original is drawn round the system *A*, *B*, *C*, *E*, in the same manner as the pattern in the manufacture, and kept stretched by means of the screw *F*. The original is then lightly coated with oil, so that the mass may subsequently easily be removed. In the pan *L* is the mixture for the pattern, consisting of glue, glycerine, and pyroligneous acid. The mass is applied in the same manner as described above. The surface is then hardened by treatment with acetate of iron, and sufficiently dried.

The mixture for the manufacture described above consists of glue, colour, glycerine, linseed oil, soap, gallotannic acid, and chromic acid.

CHAPTER XXVII

GLUE-MAKERS' WASTE

Utilisation of the Fat obtained in Glue-Making to obtain a Solid Lubricant.—The raw material from which glue is made is generally waste matter, such as the waste of slaughter-houses and tanneries, old shoes, rabbit and hare-skins from which the hatmakers have stripped the fur, also cat and dog-skins, ox feet, calves' and sheep's feet, sinews and entrails. These substances receive a preliminary treatment with lime, in order to remove particles of blood and flesh, which would hasten decomposition and give the glue a dark colour, and also in order to saponify the fat. For this purpose the substances are treated in large pits or tanks with thin milk of lime for fifteen to thirty days, during which time the lime is often renewed. When this has been done, the adherent lime is removed from the animal substances, which are worked up for glue.

This residue, which, in addition to lime, contains the fatty parts of the substances treated, saponified by lime, is known as "glue fat." The cheap lime soaps, which can be bought under this name, may with advantage be used in preparing machinery greases. The process is as follows:—The lime soaps are placed in a pan, which they half fill, and then heated over a gentle fire until they melt. The fire is then gradually increased and the boiling continued until the mass has been so far boiled down that a test, brought on to a sheet of glass, can be drawn out into long threads on touching with the finger. In order to hit the right point, tests must frequently be taken during the boiling, since, if the mass were to thicken too far, it could not be used, and would be wasted. Thick mineral oil is now added gradually in small portions, and with continual stirring, until the desired con-

sistency, which is tested by taking out portions on to a glass plate, is reached. The mass is then run into a tub and stirred until nearly cold.

Since glue fat contains so many impurities—dirt, hairs, etc.,—it must previously be cleaned, which is best combined with the melting process—the dirt swimming on the top being skimmed off, and the deposit which collects at the bottom removed.

CHAPTER XXVIII

ILLUMINATING GAS FROM WASTE, AND THE BY-PRODUCTS OF THE MANUFACTURE OF COAL-GAS

Utilisation of Waste Products in making Illuminating Gas. Gas from Suint (*Dingler's Journ.* 195, pp. 175, 216, 517).—Of the waste products containing fat—bones, oil-cake, soap waters—the latter especially, produced in washing the grease out of wool and the gum from silk, may be used with advantage for the production of an illuminating gas. It contains unaltered fat, oleates and stearates of soda and lime, and also organic matters from the wool and silk. The fatty acids are separated from the suds either by inorganic acids, or they are combined with lime and the product subjected to distillation. (*a*) *Distillation of the separated fatty acids.*—According to Knapp, at Rheims 300 cwt. of wool-washing suds are mixed with 2 per cent. of sulphuric acid or 4 per cent. of hydrochloric acid and equal quantities of water, and allowed to stand for twelve to eighteen hours, when the impure greyish mass of fat which has risen to the surface is mechanically cleaned, melted in a copper pan, and whilst fluid, treated again with 2 per cent. of sulphuric acid. The clear oil obtained is used for soap-making, and the black, solid, fatty residue employed for producing gas. The tar which separates in the distillation is always again used to dissolve the solid residue. (*b*) *Distillation of the separated lime soaps.*—At the spinning-mills in Mühlhausen and Augsburg, according to Altgelt, the water containing the sweat of the wool and the used soap, is allowed to stand with milk of lime for twelve hours, the clear liquid drawn off, the deposit sieved through coarse linen cloths, and the mass which passes through allowed to stand for six to

eight days in a cellar until it forms a pasty mass, which is then cut up with a spade into rectangular lumps of the size of half a brick. These lumps are dried for several weeks on frames, and the dried mass, which is termed *suinter*, subjected to distillation. A gas is obtained which it is not usual to purify, and which has three times the illuminating power of coal gas. The soap liquors in which silk has been boiled, according to Jeannency, are heated with slaked lime to 70–75° C., allowed to clarify during two to three days, the clear liquor decanted off, the deposit filtered, spread out to dry in the air, and then used for gas-making. One hectolitre (22 gals.) of the soap liquor requires 0·75–1·5 kilo. (1·68–3·37 lb.) of quicklime, and gives 1200–1600 litres (42–56 cub. ft.) of gas. The gas is not purified or washed, but is taken direct from the retorts to the gasometer.

Illuminating Gas from Wool Waste.—Liebau, of Magdeburg (*Dingler's Journ.*, 184, p. 380), employs wool waste from spinning mills either alone or, better, mixed with coal to produce illuminating gas. A layer of coal 52 mm. deep (2 in.), then a layer of wool waste 157 mm. (6·3 in.) deep, and finally a little more coal, are brought into the scoop used for filling the retorts, and the contents quickly emptied into the retort, which is at a low red heat. With a retort of 30 kilos. (67 lb.) capacity the distillation is finished in one and a half to two hours. Fifty kilos. (1 cwt.) of waste produce 21·63 cub. metres (800 cub. ft.) of gas, whilst the coal also gives about 17 cub. metres (630 cub. ft.) of gas per 50 kilos. (1 cwt.). The carbonic acid is to be removed by purifying. It is advisable to use the wool waste mixed with coal rather than alone, since a saleable coke is produced, the cheaper clay retorts can be used, and the purification is also less expensive.

Illuminating Gas from Beet Molasses, Wine Lees, and Grape Marc (*Deutsche Industrie-Zeit.*).—From 100 kilos. (2 cwt.) of dried, or 111 kilos. (250 lb.) of crude molasses, Stammer has obtained 61·8 cub. metres (230 cub. ft.) of unpurified illuminating gas. The residues from the maceration of dried beet, after pressing and drying, give 24·72–37·08 cub. metres (916–1375 cub. ft.) of gas per 100 kilos.

(2 cwt.). The gas contains 23–24 per cent. of carbonic acid. Three kilos. (6¾ lb.) of 10 per cent. ammonia liquor or 1·2 kilos. (2·6 lb.) of sulphate of ammonia are also produced.

The grape marc, which has already been used for producing tartaric acid and marc brandy, may further be used with profit for gas-making (*Die Verwerthung der Weinrückstände*, A. dal Piaz, Vienna). No other appliances are necessary for producing gas from the grape marc than those used for coal- or wood-gas. Grape marc may therefore be treated in any well-arranged coal-gas works. The by-products obtained by condensation from the gas produced by grape marc are essentially the same as those obtained from wood-gas, viz. acetic acid, ammonia solution, and a mixture of hydrocarbons similar to wood-tar, which may again very well be utilised for the preparation of creosote, photogene, and paraffin. It is absolutely necessary that the marc used for producing gas should be free from alcohol, *i.e.* only marc can be used from which brandy has been prepared or from which the alcohol has evaporated by long lying in the air; it must also be air-dried, and should not be mouldy. The marc, as it comes from the brandy distillery, is pressed in order to remove the greater part of the liquid. It is then dried, for which purpose it is pressed into moulds, which may be either rectangular boxes, open at top and bottom, or circular rings; rectangular bricks or round cakes are thus produced in the same manner as the bricks of tan-bark. The bricks or cakes are quickly dried in the open air on hurdles or frames. Drying the marc by the aid of artificial heat is not advisable, since if the drying be carried only a little too far, not only is much less gas obtained, but also gas of lower illuminating power. The dried bricks may be kept for years before use, if they are piled above one another in open layers so that the air can always pass between them. The retorts are charged in the same manner as with coal. At each charging 20–50 kilos. (42–112 lb.) of the marc bricks may be introduced, according to the size of the retorts, so that five or six-tenths of the internal volume is occupied. The quantity of marc cakes required for one charge is distributed in a charging scoop, of the same length as the

retort and of semi-circular section. The scoop is then quickly pushed into the retort and turned over, so that its contents are emptied on the bottom. The empty scoop is rapidly withdrawn and the retort lid, the edge of which is well smeared with clay paste, screwed on. When the retort is closed, a violent distillation and evolution of gas take place as soon as the retort is sufficiently heated. As in making wood-gas, a charge of grape marc is completely gasified in two hours at the most, whilst coal requires five to seven hours before it is entirely exhausted. The treatment of the grape marc has thus the advantage over that of coal that a large quantity of gas can be rapidly made without large furnaces and gasometers being necessary. The gas is also of greater illuminating power than ordinary coal gas, but particular care must be taken that the grape marc used is well air-dried, and quite free from alcohol and mould. If the intention in gasifying grape marc is to obtain good Frankfort black, then no higher temperature should be used in the distillation than is required to maintain the retorts at a dark red heat. Thus it is advisable to use for heating the retorts or gas furnaces, in place of the usual coke, a fuel of less heating power, such as turf, lignite, or wood, especially when coke is the more valuable. When grape marc is gasified with the retorts at a dark red heat, a coaly residue of a dull, deep bluish-black colour is obtained, which produces Frankfort black of a very fine shade. If, on the contrary, the retorts are almost at a white heat, the residue is dark grey and shining like graphite, to which when ground it is quite similar; it may be used ground in linseed oil as "furnace black," a greyish-black paint of good covering power.

After the distillation has continued one to two hours, according to the size of the retorts, the evolution of gas slackens, the gasification of the charge may then be regarded as finished and the hot residue at once withdrawn from the retort. When the retort lid is opened, the gases issuing from the retort are lighted, as in making coal gas, in order to avoid an explosion. The withdrawal of the red-hot charge must, similarly to the charging, be as rapid as possible, so that the

residue may not be partially burnt, when it would be contaminated by a fine white ash.

It is therefore necessary to use a broad rake in emptying the retorts, to rake the contents into a sheet-iron box with a well-fitting lid, placed below, and at once to close the box with the lid in order to exclude the air. It is, however, better and simpler partially to fill the box with water, so that the glowing contents of the retort are quenched immediately they are drawn.

In making gas from grape marc no other condensing apparatus is required than with coal- or wood-gas; as with the latter, dry lime is used for purifying. The gas lime obtained in the purification, and also the other by-products of the condensation, may be utilised in the same manner as those obtained from wood-gas or coal-gas. Experiments made at the Grünstadt gasworks have shown that when the retorts are almost white hot, from a 50 kilo. (1 cwt.) charge of completely air-dried grape marc about 17·5 cub. metres (648 cub. ft.) of gas are obtained of a considerably higher illuminating power than ordinary coal-gas. At a dark red heat the yield of gas is smaller—from a 50 kilo. charge about 15·6 cub. metres (577 cub. ft.) of gas of the same illuminating power as ordinary coal-gas.

The grape seeds, separated from the marc and dried, in consequence of the oil they contain, give almost twice as much gas as the marc, and a gas far surpassing gas from marc in illuminating power. The carbonaceous residue obtained in gasifying the seeds also gives Frankfort black. In gasifying grape marc the residue, *i.e.* the Frankfort black, amounts to one-quarter of the weight of the dried marc; it is possible to reckon with certainty on obtaining 20–25 per cent. of grey or black pigment.

Illuminating Gas from Fusel Oil (*Zeits. d. Ver. f. Rübenzücker-Industrie*).—The fusel oil from potato spirit is generally preferred in the manufacture of perfumes to fusel oil from beet or molasses distilleries, since it contains more amyl alcohol. H. Briem recommends that this by-product of the molasses distillery, which has little commercial value, should be used for preparing illuminating gas. The iron pipe

required for decomposing the fusel oil has a diameter of about 10 cm. (4 in.) for lighting 100–200 burners; it is laid in the fireplaces of the furnaces used for evaporating the wash.

Illuminating Gas from Sewage Deposits and Excrement.—The first experiments with this object date back to 1827, when Reimann, of Berlin, made illuminating gas from fæces. The price of the new gas was, however, higher than that of coal gas, and thus this method of utilising excrement had to be abandoned. Of the more modern process, two in particular are worthy of notice: the first, for the production of illuminating gas and hydrogen from sewage deposits, is due to R. G. Hickey; the second, of illuminating gas from excrement, to Sindermann, of Breslau.

Hickey has published a description of his process (*Dingler's Journ.*, 195, p. 378), according to one modification of which hydrogen, for illuminating purposes, is obtained by passing superheated steam over sewage deposit, heated in a retort. Another modification consists in producing a gas for lighting and heating from sewage deposits. Hickey intended to make excrement of all kinds innocuous in this manner, by carbonising it in closed retorts, and using the residue in the retorts for removing the smell of fresh matter before its introduction into the apparatus, and also, together with the distilled products, as manure; the gas was to be used either for lighting or heating, or for both purposes together. The process differs somewhat according to the result desired. For the transport of the sewage deposit to the gasworks, buckets of circular or elliptical section are used; they narrow at the top to a neck from three to four inches wide, which is filled with the ignited excrement, and may also be closed by a water-lute, to prevent the escape of smell. The fluid constituents are partially removed from the matter before it is brought into the retorts, and are also made odourless by means of the ignited residue. For this purpose the mixture is brought into a cylinder with a perforated bottom, covered by a layer of ignited residue. When full, a tight-fitting plate is laid on at the top, and then, by a screw mechanism, a gentle pressure is applied, which expresses a large part of the liquid in a completely innocuous condition.

The retorts in which the pressed matter is heated are either of clay or wrought-iron; they are best ⊓ - shaped, and have at each end an opening, closed during use by a tight-fitting lid, smeared with clay; the retorts are laid at an angle of 15–20°. If the gas is only to be used for heating, the gases and vapours are allowed to rise through pipes, and then through a water-tank divided into several compartments; in this manner they are completely washed, and the ammonia salts mostly separated. The gas then goes through a condenser, and thence into a small gasometer, from which it passes through a pipe, furnished with a regulating valve and ending in a head provided with slits, to the furnace, where it is burnt with the addition of partially carbonised excrement. If the gas is to be used for lighting, it must be more carefully purified. It leaves the retort, as does coal gas, to enter the main pipe, which must be of relatively large size; it goes thence to the condensers, in which the ammonium and other salts are removed by repeated passages through water. It then goes through a scrubber—a cylinder filled with bricks, stones, charcoal, or large lumps of the retort residue,—and finally through a dry purifier, which contains alternate layers of lime and retort residue, spread on frames one above another, and which it leaves for the gasometer.

If the gas produced in the retorts is to be used for heating, and, at the same time, hydrogen gas is to be made for lighting purposes, the gas which is first evolved is conducted away, as in the first case; as soon as the evolution of gas has ceased, the connection with the pipes leading to the condenser is cut off by a tap, and then superheated steam, from a small boiler built in the furnace, and which may be fed with the liquid expressed from the excrement, is passed through a perforated pipe over the red-hot residue in the retort. Pure hydrogen and carbonic acid are formed almost entirely; the latter is removed in a dry lime purifier, whilst the hydrogen is burnt in an Argand burner, provided with platinum gauze.

The gas produced from human excrement burns with a very bright flame. The residue remaining in the retorts is a black powder, quite odourless, which is very suitable for removing the odour from solid and liquid sewage matters, and,

like the deposit in the condenser, produces an excellent manure. From the liquid condensed in the main pipe, by evaporation with hydrochloric acid in pans on the top of the furnace, sal-ammoniac is obtained in large quantity.

A very favourable report on Sindermann's process was given in 1875 by the Breslau Common Council, which had appointed a special commission to examine it. The fæces are brought in small quantities—with the apparatus then used, 2–3 kilos. ($4\frac{1}{2}$–$6\frac{3}{4}$ lb.)—every fifteen to twenty minutes into a heated retort. 100 kilos. (2 cwt.) of fæces require 50 kilos. (1 cwt.) of coal, and produce 7·8–9 cub. metres (290–333 cub. ft.) of gas, and with the addition of 1 kilo. ($2\frac{1}{4}$ lb.) of iron turnings, 24 cub. metres (888 cub. ft.) of gas. In addition, there are said to be obtained—6·66 kilos. (15 lb.) of coke, which may be used as manure; 3·33 kilos. (7·5 lb.) of tar; 3·33 kilos. of fat, which is used for protecting the collecting barrels; and large quantities of ammonia liquor.

The process just described can hardly exist in actual practice; in a critical examination by Fischer, in *Dingler's Journ.*, 217, p. 425, it is entirely rejected. Therefore it is here mentioned that, according to Gintl, fæces contain an average of 92·5 per cent. of water and 1·6 per cent. of ash. Thus, in order to obtain 5·9 kilos. of organic matter, from which gas can be made, 92·5 kilos. of water have to be evaporated, which again must be condensed in a gigantic cooling apparatus. Whilst a coal-gas works consumes barely half the coke it produces from the coal, in treating fæcal matter large quantities of fuel must be purchased. The production of larger quantities of gas by an addition of iron turnings can, at least in substance, only result from the decomposition of water by heated iron. Now

$$\begin{array}{cccc} 3Fe + 4H_2O & = & Fe_3O_4 & + 4H_2 \\ 168 \quad 72 & & 232 & 8 \end{array}$$

therefore 168 kilos. of iron give 8 kilos. or 89·5 cub. metres of hydrogen; thus 1 kilo. of iron, even when it is completely transformed into Fe_3O_4, can only give 0·5 cub. metre. Sindermann's statement, that an addition of 1 per cent. of iron turnings can increase the yield from 8 to 24 cub. metres,

must therefore rest on error. The gas liquor will, as a rule, barely contain 0·5 per cent. of ammonia, and is therefore far more difficult to utilise than that from coal. Also, the tar and the fat apparently obtained seem to have a low value. Since, further, the tending of the apparatus requires relatively much labour, the process cannot be profitable.

Treatment of the Used Laming's Mixture of the Gasworks.—For many years the chemical works of Kunheim, Berlin, have worked up the Laming's mixture from gasworks (F. Beilstein, *Die chemische Grossindustrie; Dingler's Journ.*, 211, p. 76). The mixture of ferric oxide and lime, which are the essential constituents, soon absorbs a large quantity of ammonia, sulphur, and cyanogen compounds. By washing with water the ammonia salts are extracted and isolated. The residue, when decomposed with lime, give a soluble double cyanide, from which yellow prussiate of potash may be obtained by precipitation with potassium sulphate. The insoluble portion, when roasted, gives up the whole of its sulphur, which is utilised in vitriol chambers; there remains ferric oxide in a condition very suitable for the purification of gas. In addition, ammonium sulphide, and, by direct treatment with hydrochloric acid, Prussian blue, may be prepared.

Recovery of Sulphur from Laming's Mixture.—The sulphur deposited in the mixture may either be extracted with carbon bisulphide, or may be industrially utilised by burning the mixture in the sulphur burners to sulphur dioxide, when 1 ton produces 1¼ ton of sulphuric acid (*Dingler's Journ.*, 196, p. 372). G. Pelouze recommends to utilise the solubility of sulphur in coal-tar oils for its extraction from Laming's mixture (*Dingler's Journ.*, 196, p. 372).

Ammonia from Gas-Liquor.—According to a patent of Braby and Baggs, ammonia is obtained from gas-liquor by adding caustic lime, heating in a boiler to a temperature between 40° and 100° C., and then forcing air through a pipe reaching to the bottom of the boiler, the air being further distributed by a perforated bottom. The air, carrying ammonia and a little steam, leaves the upper part of the boiler and passes into a receiver filled with water, hydrochloric or sulphuric acid. At Deptford, where this process

was employed, the air, laden with ammonia, first goes through a lime purifier containing milk of lime kept in constant motion by a stirrer, and then through a cooled coil to the receiver, which consists of three vessels. Two of these vessels are one-third filled with cold water, the last is best filled with a strong solution of ferric chloride, which is converted into a solution of sal-ammoniac with precipitation of ferric oxide, which, after ignition, can be used as a painter's colour.

Utilisation of the Ferric Oxide used for the Purification of Coal-Gas.—Various methods have been proposed. According to an English patent of H. Grüneberg, the spent purifying mass, after extraction with water and alkali, is treated with hydrochloric acid in order to separate part of the sulphur, and is then converted into Prussian blue by means of a ferric salt and bleaching powder. (*Dingler's Journ.*, 227, p. 212.)

Gerlach proposes to grind the purifying mass fine, and then to extract first with water, next with caustic soda solution. From the last extract, sulphur and cyanides are precipitated on the addition of acid to slight acid reaction; ferric chloride is then added to the solution drawn off from the precipitate and filtered if necessary. The residue remaining after the two extractions yields its sulphur on distillation in an iron or clay retort in a current of superheated steam. The extracted and desulphurised mass is converted into colcothar by heating with access of air.

P. Spence uses the following process:—The spent iron oxide is first washed with water in a suitable tank, in order to remove any ammonia compounds present. The mass is then again dried in the air, a quantity of quicklime equal to half the weight of the oxide is slaked and the dry hydrate intimately mixed with it. The mixture is lixiviated with warm water—not above 70° C.—in iron vessels with double bottoms. The solution when slightly acidified gives, with ferric chloride, a precipitate of Prussian blue. This precipitate is used as such, or employed to prepare yellow prussiate of potash. After the ferric oxide has given up all the potassium ferrocyanide, it is heated in the same vessels with water to boiling. A solution of calcium polysulphides

is obtained, from which hydrochloric acid precipitates sulphur. The ferric oxide thus extracted is again used for purifying coal-gas. (Eng. Pat. 4118; *Chem. Centralblatt.*, 1879, No. 28.)

Prussian Blue from the By-Products of the Manufacture of Coal-Gas.—According to the English patent of Valentin, ferric hydroxide, which has been used for purifying coal-gas, is washed with water, digested with magnesia or chalk at a high temperature, and then extracted with water. The pale yellow, somewhat alkaline solution contains calcium or magnesium ferrocyanide; on the addition of a little acid and an iron salt, it deposits fine Prussian blue.

Ammonium Sulphate from Gas-Liquor is made by P. St. Brown, in Ireland (Post's *Zeits. f. d. chem. Grossgew.*, IV. 1), by incompletely neutralising with sulphuric acid, thus leaving the liquor rather alkaline, and evaporating in iron vessels until ammonium sulphate crystallises out. The product is somewhat impure, and can only be purified by complex and repeated recrystallisations.

H. Krätzer has accordingly recommended a new method for the preparation of ammonium sulphate, which has been adopted by several chemical works, and which produces an almost completely pure salt. A little lime is added to the gas liquor in order to decompose the ammonium salts it contains; a rapid current of steam is then passed through the mixture. The whole of the ammonia is expelled, and when conducted into a water-tank gives at once ammonia solution of any required strength, from which, by neutralisation with sulphuric acid, a product can be obtained far more pure than by previous methods. 100 parts by weight of gas liquor give 1–2 parts of the salt. This method is particularly to be recommended to those works which make ammonium sulphate for use as a fertiliser, since the product is free from sulphocyanides and hence is harmless for agricultural purposes. Krätzer showed, as a result of many experiments undertaken when director of the Agricultural Institute at Brandis, that sulphate of ammonia, made by older methods, generally contains potassium sulphocyanide, and then injures the crops instead of improving them.

Simultaneous Utilisation of Coke Dust and Gas-Tar.—In large gasworks coke dust is produced in considerable quantities; it has hitherto been utilised only by a somewhat laborious process of little profit. It is mixed with small quantities of coal-tar, or tar freed from its volatile constituents by distillation, or even with loam or clay, and moulded into briquettes by special machinery. The briquettes are a good fuel, but they require to be made by powerful machinery, and are consequently expensive. Coal-tar, in consequence of its chemical composition, is by itself a valuable raw material for the production of illuminating gas, but all attempts to utilise it in this manner have failed owing to various technical difficulties. The engineers of the Paris Gas Company have proposed to utilise coke dust and coal-tar by the simultaneous production of a good fuel from the former and of illuminating gas from the latter by-product, and have obtained a patent for the process.

In any simple mixing apparatus, 50 parts by weight of tar are well mixed with 40 parts of coke dust, brought into the retorts by means of the ordinary semi-cylindrical scoop and heated for three or four hours at a temperature of about 1200° C. After the lapse of this time, the distillation of the admixed tar is finished, and the solid coked residue has sintered with the coke dust to a solid mass, which has only to be cooled by water or steam and broken up, in order to yield an excellent fuel, burning readily in a grate with a slight draught, and especially suitable for household use, etc. (Armengaud's *Publication industrielle*, 22, p. 491; *Dingler's Journ.*, 219, p. 470.)

Utilisation of Lignite Coke.—It is well known that, in the dry distillation of lignite, coke is obtained in considerable quantities as a by-product which is difficult to utilise. Formerly it was only applied as road material, but later (Post's *Zeits. f. d. chem. Grossgew.*, III., 1) it has begun to be used in the so-called ashpit stoves. The *Wochenschrift für Oel- und Fettwaarenhandel* calls attention to other uses for this by-product. Coleman has (*loc. cit.*) made successful experiments with a view to using lignite clinker for disinfecting the latrines of two large hospitals in Glasgow.

Two parts of the contents of the latrines, mixed with 1 part of bone black or powdered lignite coke were temporarily, when mixed with an equal quantity, permanently deodorised. Coleman accordingly proposes to use lignite coke for disinfecting the contents of the sewers of towns and to utilise the product as manure.

There have also been several communications in this connection to the *Zeits. f. Paraff.* in regard to the utilisation of the lignite coke of oil distilleries for the same purpose, for which equally favourable results have been obtained. However, more may be expected of the application of lignite coke as fuel, if only suitable fire grates can be constructed for the purpose.

(In regard to working up the tar, which is an industry in itself, to describe which would be to overstep the limits of this work, the reader is referred to the excellent and practical work of Dr. J. Bersch, *Die Fabrikation der Anilinfarbstoffe*, Hartleben, Vienna.)

CHAPTER XXIX

MEERSCHAUM

Utilisation of Meerschaum Waste.—The dust and cuttings produced in making genuine meerschaum pipes are carefully collected and used to produce imitation meerschaum, which is considerably inferior to genuine in quality.

According to T. Urban (*Dresd. Gewerbevereins-Zeit.*), the waste from genuine meerschaum is stamped in a barrel with water, the coarse mud brought into a mill, consisting of two stones, one lying close on the other, and here ground still finer. The paste is then rubbed by hand through a linen cloth stretched over the top of a tub. The fine material is now well boiled with linseed oil with the addition of alum. The linseed oil gives the imitation meerschaum the peculiar shine of the real; the alum takes the place of a binding medium. When the boiling is finished, the meerschaum is brought into moulds and allowed to lie in the stove until the adherent water has completely evaporated and the mass has acquired about the consistency of soap. When this stage is reached, it is easy to bring it into any required mould and to shape it with the knife.

When the shape of the pipes is roughly obtained, the turning is completed on the lathe. The pipes are then dried in a stove at 75–87° C., and subsequently boiled in melted suet. After cooling, they are rubbed with grass (*equisetum*). After rubbing, the best qualities are dipped in boiling spermaceti or wax, which causes them to colour more quickly when used, and at the same time gives the meerschaum a better polish and greater hardness. Meerschaum pipes burnt black inside and out are in equal demand with the white pipes; they are distinguished by a very fine polish. The blackening is obtained by placing the white pipes for fifteen to thirty minutes in boiling linseed oil, and then holding them over burning pine sawdust until they are coloured black or dark, after which they receive the real polish.

The red meerschaum pipe, or oil pipe, is a lower quality, which is obtained by first boiling the cut pipe in fat, then turning, polishing, and finally again boiling. The red colour is produced by adding dragon's blood to the linseed oil; according as the colour is to be dark or pale, more or less dragon's blood is added. Carmine, gamboge, and alkannet are also used as colouring matters.

The imitation pipes—made from waste meerschaum—are also carved. The carving is done in the half-boiled state, and the pipe is finally again boiled.

J. S. Hyatt, of Paris, has discovered a process for the *preparation of a plastic substance from meerschaum waste*, nitrocellulose, and camphor (*Ackermann's illustr. Gwb.-Zeit.*). In making certain articles, the shape and size of which considerably exceed those of the crude lumps of meerschaum, it is evident that both real meerschaum and the waste may be used, since the cost of treating and moulding the composition for this purpose is considerably less than that of the system now used—cutting the articles out of the solid. The process used is as follows:—In the first place, the meerschaum waste is in any manner converted into a fine powder. Next a solution is made, containing 5 parts by weight of nitrocellulose and 3–5 parts of camphor, by adding a quantity of ether (about 3 parts) and alcohol (about 1 part), or other liquids in which nitrocellulose dissolves, sufficient to produce a viscid mass. The proportions of the four constituents here given have yielded excellent results in practice. They are not, however, to be regarded as absolute, and may be varied within certain limits according to the nature of the raw material and of the object to be produced.

Powdered meerschaum is added to the above solution in about the proportion of 100 parts of meerschaum to 5 parts of the dissolved nitrocellulose. The whole is then thoroughly mixed in any desired manner, and the excess of solvent allowed to evaporate by natural or artificial means. The mass is next thoroughly powdered; the powder is the substance actually used in making a large number of articles. For this purpose it is filled into moulds made of metal or other material, and heated to a temperature of 100–120° C.

CHAPTER XXX

MOLASSES

Utilisation of Molasses.—Molasses are used in the manufacture of spirit. The value depends on the percentage of sugar: in order to estimate the value the molasses are diluted, say to 18° on an ordinary saccharometer, the solution mixed with an excess of yeast at 24–25° C., brought into a warm place, and allowed to ferment. If now the saccharometer shows 3° unfermented, there was in the 18° solution apparently (since the spirit present in the fermented liquid affects the saccharometer reading) 15° of sugar; thus in molasses which showed 45° originally, 37·5°. In order to find the true amount of sugar, the fermented liquid is boiled, the alcohol driven off, and the loss in boiling replaced by distilled water; a polariscope reading is again taken, when the percentage of sugar indicated by the instrument is the true unfermented. For example, if the saccharometer shows 19° before fermentation, in the unboiled fermented liquid 4°, in the boiled fermented 5½°, then there are really 13½° fermentable. (*Populäres Handbuch der Spiritus- und Presshefe-Fabrikation*, A. Schönberg, 4th ed., Vienna, Hartleben.)

Molasses consist of sugar, salts, nitrogenous and nitrogen-free compounds, alkalis, and alkaline earths. The latter hinder fermentation. In order to remove the condition which retards fermentation, 0·5–1·5 per cent. of sulphuric acid, as required, is added to the molasses. Molasses are not really mashed, but they are somewhat diluted in the mash-tun, according to their thickness, and the sulphuric acid, previously diluted with water, added. The mixture is then heated until the sour smell disappears. Acid is only to be used when the molasses have an alkaline reaction; if this be

the case, acid is added until blue litmus paper is coloured a weak wine-red. It is advisable to add the water in the mash-tun, and to prepare the liquid there, for if the water were added in the fermenting vat there would be difficulty in stirring the liquid. The molasses must be intimately mixed with water; and since cold molasses mix with water with great difficulty, it is advisable to heat whilst mixing in the mash-tun. A cooler is necessary in molasses-works. Whether sulphuric or hydrochloric acid is chiefly used depends on the success with which the liquid ferments. Generally, after neutralisation with sulphuric acid, 1–2 lb. of hydrochloric acid are added to a tun containing 90–100 buckets (say 300 gals.). Many samples of molasses require no hydrochloric acid; those which require it are generally the produce of works using beet from wet, heavy soil, or from uncultivated ground. As a rule, the quantity of hydrochloric acid is 5–10 per cent. of that of the sulphuric acid.

An intense green malt yeast is generally used for molasses wort, to which beer yeast or pressed yeast must be added every day, since molasses worts have a very heavy fermentation; 10 per cent. of yeast is employed as a rule. It is also very advantageous to initiate a very strong preliminary fermentation. The wort acquires, soon after adding the yeast, a layer on the top 3–4 inches thick, below which, if the treatment has been successful, a very intense fermentation proceeds. The fermenting vessels are filled to within 2–3 inches of the top, and the wort pitched at 30–44° C.

If beet juice is available, it is used for diluting the molasses. If a vat does not come properly into fermentation, it is assisted by an addition of beer yeast or fermenting wort. Care should be taken that the fermenting room is always at a temperature of 18–20° C. Since these worts rapidly cool, and ferment with difficulty, it is necessary to have one or two stoves in the room, so that it can be warmed during the winter months.

The spent wash of country distilleries is used as food for cattle, partly mixed with other materials; it is also used as manure. In large distilleries the wash is evaporated in the

"thickening pans," and then brought into calcining furnaces built like black-ash furnaces. As a rule three furnaces are arranged in steps—one below the other; the wash is allowed to flow from one to the other, being carbonised in the last. According to the quality of the molasses, 11–12 per cent. of carbonaceous residue is obtained, containing about 50 per cent. of potassium carbonate. The free acid in the wash is frequently neutralised with lime; the sulphate of lime is then allowed to deposit, and the wash, after evaporation in the pans, brought into the above-mentioned calcining furnace in order to destroy the organic matter.

When given in large quantities to cattle, molasses wash causes sickness.

If potatoes are to be worked together with molasses, it is most convenient to mash the two together, and to mix them in the inverting vessel. The prepared molasses wort is run into the inverting vessel during the grinding of the potatoes. If, however, there is no vessel in which the molasses wort can be made, it is done in the inverting vessel, and the ground potatoes added. The best proportion is 25 cwt. of potatoes to 2·5–3 cwt. of molasses of 42°. The manipulation is the same as with potato worts, except that the yeast must be increased in proportion to the molasses. With these proportions the wash may be used as fodder without fear.

C. Vincent has introduced a **process for the more rational utilisation of vinasse—the residue left after distilling the alcohol from fermented beet molasses** (*Chemical News*). Formerly the vinasse was calcined in open hearth furnaces for the recovery of the potash salts. The idea of utilising the gaseous compounds produced in this process had been mooted years ago, but has only recently been realised by Vincent. In this process the calcination is performed in cast-iron retorts; the products of distillation, which condense at the ordinary temperature (tar and ammonia water), are caught, whilst the gaseous products are lead away to heat the retorts. The ammonia water is practically similar to that obtained in making gas by the dry distillation of coal, but contains, in addition to the ordinary constituents, methyl alcohol, methyl cyanide, methyl sulphide,

and, what is particularly remarkable, a large quantity of trimethylamine salts.

The aqueous alkaline product of the distillation is, according to a further account in the *Pharmaceutische Zeitung*, first over-neutralised with sulphuric acid, and then distilled. Methyl alcohol comes over; on cooling, ammonium sulphate crystallises from the residue. The mother liquid contains much trimethylamine sulphate.

Trimethylamine has at present no particular commercial value; it is, however, very suitable for the production of methyl chloride. When trimethylamine hydrochloride is heated, it decomposes into ammonia, free trimethylamine, and methyl chloride. The decomposition begins when the boiling-point of the concentrated mother liquor of the trimethylamine salt has reached 260° C., and is complete at 325° C. The gaseous products are conducted through hydrochloric acid, which retains the alkaline compounds; after sal-ammoniac has been separated from this solution by evaporation and recrystallisation, the solution is again subjected to distillation. The methyl chloride which passes over, after purification by washing with dilute caustic soda solution, is dried by strong sulphuric acid, and pumped into iron or copper cylinders closed by screw-cocks. The gas is liquefied by pressure in these cylinders, in which state it is sold.

Methyl chloride has two technical applications; it is very suitable for the production of "artificial cold," and it is used in manufacturing methylated aniline dye stuffs, which have previously been very costly owing to the use of methyl iodide. When methyl chloride evaporates, its temperature falls to −13° C.; but if the rate of evaporation be increased by the passage of a current of dry air, the temperature sinks down to −55° C. Methyl chloride is thus an excellent material for ice-machines.

At the large distillery of Tilloy, Delaume, & Co., at Courrières, which was managed by Vincent, 90,000 kilos. (90 tons) of molasses were treated daily; 25,000 litres (5500 gals.) of pure alcohol of 90° Gay-Lussac were obtained, leaving 40 tons of vinasse, which yielded 10,000 kilos. (10 tons) of potash salts, and as condensation products,

1600 kilos. (32 cwt.) of ammonium sulphate, 100 kilos. (2 cwt.) of methyl alcohol, and 1800 kilos. (36 cwt.) of concentrated mother liquors of trimethylamine salts; the vinasse also yielded 4000 kilos. (4 tons) of tar, from which 360 kilos. (7¼ cwt.) of oil and 2000 kilos. (2 tons) of ammonia water were distilled off.

CHAPTER XXXI

METAL WASTE

Recovery of Brass Waste.—In order to separate mixed filings, it was usual to carry away the iron and steel by means of a magnet held in the hand, when the brass filings remained. Vavin, a French engineer, has constructed a machine to shorten this tedious operation (*Engineering; Dingler's Journ.*, 197, p. 18). It has already been tried in practice, and is made by Cail & Co., of Paris.

This machine consists of two drums, rotating on their axes, and placed one above the other. The surfaces of the drums are covered with alternate strips of soft iron and copper. Each iron strip is suitably connected with a series of horse-shoe magnets, which are so arranged that the one pole of the magnet is in contact with one iron strip, the other pole with the next. The mixture of metal filings is contained in a hopper with a vibrating foot, from which the filings fall in a stream nearly as wide as the drum. The iron filings are attracted by the iron strips of the drum, which are made magnetic, and are carried away by them until they are swept off into a receiver by a revolving brush. The brass filings and a portion of the mixture of the two metals fall upon the second drum, which is similar in construction to the upper drum, but is so placed that the iron and copper strips are in such a position to those on the upper drums that strips of different metals always come into the same vertical plane. Whilst now the brass filings fall straight off, the separating process is completed by the iron filings attached to the surface being removed by the cylindrical brush on the other side and swept into the receiver. The machine is driven by hand or from shafting; it requires a floor space of only 2 ft. 6 in. by 1 ft. 2 in., and a height of 5 ft. 3 in.

The same end is attained in a very simple and economical manner at the London and North-Western Railway Works at Crewe, by a process of fusion (*Engineering; Dingler's Journ.*, 205, p. 384). The mixed iron and brass borings and the slags from brass-casting are mixed with limestone, powdered coal, and oxide of iron, or hammer scale. This mixture is heated, when the brass separates at the bottom of the fluid slags, and is run into ingot moulds.

Bronze Pigments from Alloy Waste.—The manufacturers of imitation gold-leaf employ for this purpose the waste obtained in hammering and rolling. In the older processes (*Die Legirungen*, A. Krupp: Vienna, Hartleben) the waste from the manufacture of imitation gold-leaf is ground with honey or gum-arabic solution on a slab by a muller, until a perfect mixture is obtained. The paste is brought into water, in which the binding medium dissolves, and the metallic powder, after drying, subjected to the oxidising process. For this purpose the dry powder, mixed with a little fat, is brought into a pan and heated over an open fire until the mixture has acquired the desired shade.

At the present time this process has been shortened by the use of machinery. The proper alloys to produce certain colours, the shade of which can be obtained without oxidising, are made by melting together the metals in the correct proportions. The alloys are then beaten out into very thin sheets by hammers driven by steam-power; the sheets are converted into powder by bringing them upon fine iron-wire sieves, and rubbing them through the meshes of the sieve by a wire brush. This rubbing is accomplished under a continuous addition of oil; the mass which flows away from the sieve is then brought into a grinding-machine of peculiar construction, in which the metal particles are very finely ground. The mill consists of a steel plate, thickly studded with fine blunt-pointed needles, turning over another steel plate.

The alloys are thus obtained in the form of a very fine powder mixed with oil. The mass is first brought into water, when the greater part of the oil separates at the surface; then, on subjecting the metallic mass at the bottom of the vessel to a very high pressure, almost all the oil is removed.

The variety of bronze pigment known as "brocat," consists of somewhat coarser pieces of the alloys, which are generally obtained from the leaf metal by powdering under stamps and separating the pieces of unequal size—first by sieves, afterwards by a current of air. One species of "brocat" consists only of mica waste ground to fine powder. Many bronzes are coloured by aniline dyes, which is accomplished by adding a solution of the dye in strong alcohol to the finely ground powder, and grinding intimately together. Only dilute solutions of dyes should be used, otherwise a uniform mixing of the mass can only be obtained by very protracted grinding.

Utilisation of Copper-Wire Waste.—The so-called "leonische" wire is made from the finest and purest copper. Copper, cement, silver, and gold wire is made. The first has no coating; cement wire is coated with brass by exposing the copper rods, in a furnace, to zinc vapours. The silver wire has a coating of pure silver, and gold wire, a layer of gold over the silver. Flattened wire is called ribbon wire or tinsel. When these wires are heated, the coating disappears; it sinks into the surface, and the wire then has the appearance of oxidised copper. When this wire is brought into glass tubes, and reduced in a current of hydrogen, it exhibits the finest copper colour. The waste of fine silver wire or ribbon wire may be advantageously used, according to E. Ebermayer, in organic analysis. This fine waste is very soft, fills the tubes completely, and easily transmits the current of gas. Cement wire naturally cannot be used on account of the zinc; only silver wire should be employed. The stouter wires, discoloured silver tinsel, and lump silver may be used to produce sulphur dioxide, after which the silver can be regained.

Utilisation of the Residues of Galvanic Batteries.—The copper is first separated from the zinc solution by suspending scrap zinc in the liquid. The clear, colourless filtered liquid is then precipitated with milk of lime, when a useful zinc white of fair covering power, containing gypsum, is produced. It is particularly applicable as a water colour in printing wall-papers, and in distempering. The zinc solution may also be boiled with a definite quantity of common salt,

when there are obtained good Glauber's salt, suitable for use in glassworks, where a small quantity of zinc is not harmful, and zinc chloride, which can be used for impregnating timber and sleepers, also for producing artificial marble and a good lime cement. The zinc solution, on evaporation and strong ignition, evolves sulphur dioxide, which may be used for bleaching; hard zinc oxide is left, which is admirably suited for polishing purposes.

The copper solution is always too weak to be worked up for copper salt, therefore it is best to precipitate the copper with zinc, and obtain it in the finely divided state. In order to obtain solid copper, the copper mud and granules are collected, twice boiled with pure water, mixed with charcoal, 5 per cent. of soda, and 1 per cent. of borax, and fused to a regulus in a strong charcoal fire.

In order to obtain metallic zinc from the zinc solutions, the latter are evaporated to dryness with 10 per cent. of powdered coal, and the residue calcined in horizontal retorts; the process is then the same as in zinc-works, *i.e.* the metal is distilled.

Recovery of Waste Nickel.—The following method is used in order to recover the waste of rolled or cast nickel anodes, and also the nickel sand, which gradually collects at the bottom of the cells:—The waste nickel is repeatedly washed with clean, hot water, and then boiled in dilute sulphuric acid (1 vol. of acid to 4 vols. of water), until pure water, when poured on the nickel, is no longer rendered turbid. The nickel scrap or sand is then thoroughly dried and treated with strong nitric acid. In this operation care must be used to prevent the solution from boiling over; large porcelain vessels are used. If the solution begins to crystallise, pure water is added, and the mixture heated. The nitric acid solution of nickel must contain as little free acid as possible. The nickel nitrate is dissolved in hot distilled water, and the solution neutralised with pure caustic potash, which is cautiously added, a little at a time; the mixture is then carefully filtered. The nickel bath obtained in this manner acts directly on all metals, producing a hard silver-white deposit of nickel.

CHAPTER XXXII

BY-PRODUCTS OF THE MANUFACTURE OF MINERAL WATERS

Utilisation of the Magnesite Residues.—Magnesium sulphate, the by-product of the manufacture, consists of water (of crystallisation), 51·2 per cent.; sulphuric acid, 32·1 per cent.; and magnesia, 16·7 per cent. The two last constituents are indispensable plant-foods; the magnesia, in particular, occurs in the seeds of cultivated plants—in grain, leguminous plants, etc.—in relatively large quantity. In the condition in which it is produced by mineral-water works, magnesium sulphate contains 6–7 per cent. of impurity, and is therefore, according to Prof. v. d. Goltz, almost equal to the pure salt in value for agricultural purposes. Manuring plants with magnesia, *i.e.* with sulphate of magnesia, has repeatedly been found successful. It would be advisable to mix the salt with an equal quantity of loose earth, and then to spread the mixture over the young plants, or over fields just beginning to sprout. For leguminous plants, 0·5–0·75 cwt. of magnesium sulphate is applied per acre; 1–1·5 cwt. for clover and pasture land. Strewing magnesium sulphate over stable manure or dung-heaps is also strongly recommended.

Luhmann advises the preparation of **pure magnesium sulphate from this by-product** (*Die Kohlensäure*, Vienna, Hartleben). Since the salt is not contaminated with other soluble salts, but only with small quantities of insoluble residue and finely divided calcium sulphate (gypsum), a clear concentrated solution of pure magnesium sulphate can be obtained by systematic lixiviation. This process is conducted in a number of vats, the first of which is filled at the commencement with a strong solution of the magnesite residue in water. After the white precipitate of gypsum has settled to the bottom, the clear liquor above is drawn off by a syphon, and brought into a larger collecting tank. Fresh water is added to the gypsum, and stirred with it; then, after settling,

the dilute, clear liquor is drawn off and brought into the second vat, where it is saturated with magnesium sulphate from a fresh portion of magnesite residue. In order to fill this vessel completely, a sufficient quantity of water is added in dissolving the residue. Water is again brought on to the precipitate in the first vat, and the mass stirred. After settling, the liquor of the second is transferred to the collecting vat, and the now very dilute liquid in the first, to the second; whilst in the third vat a strong solution is made from the dilute liquor, water, and fresh magnesite residue. The process of lixiviation is thus continued, fresh vats being added until the first only contains pure water, and the Baumé hydrometer sinks to 0°. The strength of the liquors is estimated by means of this hydrometer; magnesium sulphate solution, which is saturated at 15° C., has a specific gravity of 1·299 = 16·5° B. When the hydrometer sinks to 0° in the first vessel, the water is drawn off, the gypsum mud removed, and a strong solution of magnesium sulphate made in it from a fresh portion of the residue and dilute liquor; this vessel is now to be regarded as the last of the series.

The united strong solutions of the salt are evaporated in an iron pan until a skin of salt forms on the surface, or until the hydrometer stands at 37°. After the dissolved iron has been precipitated by liver of sulphur, the fire is removed from under the pan and the liquor allowed to rest for some time until it has become clear; it is then drawn off by a syphon, filtered through a strainer, and brought into a cooler, where the salt separates in small crystals in cooling. As soon as the salt has formed a layer on the sides of the cooler about 3 cm. ($1\frac{1}{4}$ in.) thick, the crystals are stirred up with a spatula, so that the crystals subsequently formed may not be too large or the mass of salt at the bottom too hard. After the mother liquor has been run off through the bung-hole of the cooler and brought back into the pan, the mass of crystals is piled up on a capacious drainer and finally dried on gratings in a drying-stove. The iron pan is then again filled with the cold saturated solution of magnesium sulphate from the lixiviating tanks, in order to prepare a fresh portion of the crystalline salt.

CHAPTER XXXIII

FRUIT

Conversion of Fruit into Jelly.—Several recipes for this process are given in the following pages (Lucas, *Die Obstbenützung. Deutsche allg. Zeits. f. Landw.*, No. 26).

Recipe of Siemens, of Hohenheim: Pears are crushed and pressed, the fresh sweet juice is boiled down to one-third of its volume in a clean copper pan, the scum removed, and the liquid cooled; it is then strained through washed flannel. Whilst this is being done, about the same quantity of apples as of pears is well boiled with only a little water. The apples have previously been washed clean, but not pared or cut. When boiled soft they are rubbed through a hair-sieve of average mesh by means of a stiff brush, in order to separate the pulp from the skins and cores. The apple pulp is then mixed with the purified pear juice in the pan, and the two boiled together under constant stirring, until the mass adheres to a spoon in large lumps and, on cooling, has the consistency of a stiff paste. A slight addition of lemon peel, cinnamon, and cloves improves the flavour. In order to give the jelly a fine colour, about 0·5 litre of bilberry or elderberry juice is added to 100 kilos. (1 lb. to 200 lb.); a few green walnuts are also suitable.

All the jelly, whilst still hot, must be brought into the vessels in which it is to be kept; these, which are previously warmed, are best of earthenware; the jelly should not again be disturbed before use. When the jars are full, it is advisable to bring them for some time into a very hot room, *e.g.* a baking oven, so that the jelly becomes covered with a very thick, hard crust. In order to protect the surface from the entry of air, the jars are to be closed by a moist bladder. They should be kept in a dry place, not too warm.

For fruit jelly, all our sweet or less acid summer and autumn pears are suitable; of apples, baking apples, "rose apples," and other varieties ripening in the autumn. There need be no hesitation in using sourish-sweet or sweetish-sour apples, but a moderate addition of sugar is requisite. It seems to be established that the jelly from pure sweet apples and pear juice keeps longer than that in which sour apples were used.

Recipe for Lower Rhine fruit jelly: Equal parts of apples and common beet are separately half-boiled, then brought under a press together and the juice squeezed out. This juice is then placed on the fire and boiled down to a thick jelly. Pears may also be used without any different treatment being necessary: the juice is brought into a condition suitable for boiling, then freed from stalks, skins, cores, etc., and evaporated until it becomes a solid mass.

Sweet apples are generally used for such apple jelly; two of the best varieties are the *härteling*, which is the most productive apple for jelly, and produces 15–18 per cent. of jelly, and the *kernling*. Neither freezes readily; they are kept through the winter on the ground in the open, later they are squeezed and boiled down to jelly. The fruit must be ripe for storing, if it is to produce an abundance of fine jelly.

Manufacture of pure beet jelly, especially valuable in poor fruit-growing districts and in districts in which there has been a bad season: The apparatus next described and the manipulations may also be used for pure fruit jellies. After the beet has been freed from the cluster of leaves, it is washed in large tubs, or, better, in rotating lathwork drums; it then goes into a round copper pan, widening out above, in which it is boiled. In order to prevent burning to the bottom, it is well to provide the pan with a false bottom of strong wire cloth. During boiling the beet must be continuously beaten and stirred to pieces. The size of the pan is arranged according to the quantity of beet to be worked. The quantity of water is 8 litres to 100 kilos. of beet (1 gal. to 125 lb.). The beet, boiled and broken down, is then shovelled with iron spades into the press tub, which is provided with a grating

or false bottom, so that the sap can readily flow away. This grating and the sides of the tub are lined with filter-cloth, which is also placed between the layers of beet pulp at intervals of about six inches; old coffee-bags may be employed in place of the filter-cloth. When all the contents of the pan have been introduced, they are covered over with filter-cloth, the wooden cover is put in position, and the vessel subjected to the action of (hydraulic) pressure. The expressed juice runs into a receiver, from which it is pumped back into the pan. The pan which was used for the first process may now be used for boiling down the juice, or, on a larger scale, a second pan may be employed, but in the former case the pan must be very carefully cleaned—scoured with sharp sand. The evaporation then proceeds regularly; towards the end it is better to add a little oil or lard, in order to prevent frothing and burning to the bottom; the fire is also slackened. The consistency of the syrup is judged by the "thread" test: it is stiff enough when a thread 5 cm. (2 in.) long can be drawn out between the index finger and thumb. A small spoonful of the syrup may also be placed in a shallow iron vessel and cooled in cold water, when the consistency may be judged. One press is required for every two pans, one of which is used for boiling the beet, the other for thickening the juice. The whole process requires on the average a period of seven working hours. The finished jelly is, as a rule, first brought into earthenware pots, and then, when it has half-cooled, into the vessels in which it is to be kept.

The utilisation of fallen fruit has been treated by a practical fruit-grower (*Praktischer Rathgeber im Obst- und Gartenbau*, 1898):—In the first place, it is not desirable to allow any fallen apples to spoil. The author collected the fallen apples in July, boiled them soft with much water, and then stirred up to a paste in order subsequently to obtain a jelly. The mass, which was obtained from quite green, unripe apples, had a very unpromising appearance; however, the requisite sugar was added. The whole process therefore was:—The unripe apples were cleaned, brought into the boiling-pan, water added to cover them, and the contents then boiled and

stirred until a fluid paste was produced. This paste was transferred to a coarse linen cloth and allowed to drain. The juice was then boiled with sugar to a jelly: 400 grms. of sugar to 1 litre of syrup (4 lb. to 1 gal.), boiled about forty-five minutes. This jelly from unripe apples has a fine red-gold colour and a good flavour; its cost is quite low—1 lb. costs not quite threepence.

CHAPTER XXXIV

THE BY-PRODUCTS OF PAPER AND PAPER-PULP WORKS

Utilisation of Waste Matters in Paper and Paper-Pulp Works.—The waste waters, produced in boiling, washing, bleaching, in the paper-machine and also in certain subsidiary operations, constitute the greater part of the by-products and require the most attention (J. B. Höhn, *Anzeiger für Papierindustrie*, 1894). Fibres of all kinds, fine suspended mineral and vegetable compounds, colours in solution, and the many different chemicals, constitute the impurities and the materials carried away in the waters. The most valuable of these substances are the fibres which originate in the various raw materials. In order to retain them, pulp-catchers of the most varied construction, settling tanks, and also filters are used. The most convenient and cheapest process consists in first allowing the waste waters to pass a good pulp-catcher and then to deposit in settling-tanks of sufficient size, of which several must always be provided, for alternate use and cleaning. They should also be so arranged that the water can rise through a considerable distance and flow away smoothly over a considerable breadth, so that everything may be deposited in the almost motionless water. The separate compartments should be emptied at not too great intervals, since, in consequence of the presence of the various organic matters in the deposit, decomposition may readily set in, which would also attack the fibres, making them lose strength, or even quite destroying them. The regained material is especially suitable for grey or dark packing paper. Since it consists of the finest fibres, it should not require the least preparation, but can at once be introduced in the paper-machine. If suitable papers are not

made, this recovered fibre can readily be sold at a good price to pasteboard-makers. In order to make it suitable for transport, it is pressed. The waste waters of cellulose works in particular carry away much good pulp, so that some arrangement of the kind described is very essential. As regards the separate divisions of the manufacture from which the waste waters are derived, the liquors of the rag-boiler are first to be considered; they contain so little fibre, that they may be excluded from the fibre recovery. On the other hand, they are very rich in nitrogen, which results from the fat, perspiration, and other matters extracted from the rags, and since they also contain lime they produce an excellent fertiliser. For this purpose they are run into a pit, from which they can be withdrawn as required. The offensive odour is removed by gypsum, which also results from by-products. These waters may also be used for moistening the heaps of fertilisers which are composed of other waste products.

The waste liquors of cellulose works are of different composition according as soda- or sulphite-cellulose is made. In the former case the usual method for regaining the soda is employed, in which the very dilute liquors, which cannot profitably be calcined, are used to dissolve fresh portions of soda. For sulphite liquors there is not yet known a really useful and practicable process of recovery or of further treatment, in order to obtain tannic acid, sugar, or other substances. They can, however, generally very well be used for manurial purposes, after the free acid has been neutralised by alkaline or lime waste, which is always simultaneously produced. This is an inexpensive process, if conducted as mentioned above for the liquors from the rag-boiler. Where it has been tried, it has been found that all the liquors could be disposed of in this manner, and thus the nuisance of the waste waters quite removed. Also this liquor, in consequence of the bisulphite and tannic acid it contains, is an excellent substitute for alum and sulphate of alumina in rosin sizing. It may also very well be used for brown and black colours.

The waters from the beating-engine are those from which it is most important to recover the fibre. Those from the

paper-machine, however, which also contain clay, size, colouring-matters, and the like, in order to utilise these substances as far as possible, are best used over again several times by taking them back repeatedly to fill the beating-engine, to dissolve starch and clay, or to the rag-boiler. Since a certain quantity of water can only take up a certain quantity of foreign matter under given conditions, it is easily seen that the loss is considerably reduced in this manner. The composition of the paper is naturally very important. With paper which is not sized, coloured, or weighted, such a mode of treatment is unnecessary. In the case of the water from highly coloured paper, which would strongly contaminate the river into which it flowed, it would be advisable to beat and wash the rags from the boiler with it, by which means the colouring matter would be partially fixed and partially destroyed. The bleaching-powder residue is also to be considered along with the waste waters. It cannot be employed as manure. It is best collected in a pit and converted by stirring with soda solution into calcium carbonate, or with sulphuric acid into gypsum, both of which may be employed as fertilisers, especially when mixed with the above-mentioned liquors, or they may be used as fillers if the residue was pure. Carbonate of lime, which is less suitable for use as a filler, may be converted into quicklime in a small limekiln.

Of less importance are the residues of rosin, colour, and china clay, but if they have accumulated or are produced in large quantity, they are mixed, allowed to dry in the air, ground with a suitable filler on an edge-runner mill, and used as a substitute for earth pigments for common papers. An excellent earth pigment, as a substitute for dark ochres and umber, may be obtained in sulphite-cellulose works by treating the burnt or spent pyrites, which consists to a great extent of ferric oxide. The material is first broken up under stamps to a hazel-nut size, the finer portion sieved off and levigated. There results a very fine, soft colour of great staining power. In view of the quality of the material, the levigating apparatus may be of very primitive construction, the cost of working consists only of the labour, and the

process is very remunerative. The larger pieces of the burnt pyrites which remain are composed mostly of unburnt pyrites and are taken back to the furnace.

The grey arsenic sublimate is to be regarded as an unimportant by-product of cellulose works; it is deposited in the gas flues, and is generally disposed of in the wash waters. This is a grievous wrong to the fish, for it is harmful at very great dilutions—far more so than all the waste liquors. The deposit is formed in the pipes in solid form, so that it can readily be collected; in large parcels it always finds purchasers. As a result of the frequent blowpipe solderings, there is gradually formed in the gas-producing apparatus a residue of cadmium, an uncommon and dear metal which, in all other branches of industry, is collected for utilisation. Finally, calcium monosulphite and gypsum separate from the sulphite liquor on standing, and also on boiling. The former can be used as a solid "antichlor," when it is converted into gypsum, or it may be mixed with the fertilisers already mentioned; also it may be converted by sulphuric acid or a warm solution of sodium sulphate into gypsum, or by soda into calcium carbonate, in which last case it is again used in preparing fresh boiling liquor; it is also used for this purpose without treatment, but since it is not finely divided, but is generally caked together and encrusted, it has not been found very suitable.

The residues of the soda recovery, *i.e.* of causticising, in the soda-cellulose works are of particular importance—in the first place, because of their great quantity. This by-product consists essentially of carbonate of lime, contaminated by soda, carbon, and other matters. It is best utilised by burning after it has dried to a certain extent; the requisite quicklime is thus re-obtained. In consequence of the large quantity to be dealt with, continuous working is necessary, and thus fresh lime is continually produced. For conversion into gypsum, the by-product is treated with sulphuric acid; the soda salt must be completely neutralised if the gypsum is to be used as a fertiliser.

Certain by-products, which do not come in contact with water, are produced in wood-pulp and cellulose factories, viz.

the waste—sawdust, bark, and other pieces of wood—obtained in cutting and dressing the wood. The sawdust constitutes an excellent fuel when burnt in specially constructed grates; the bark is used as litter for cattle, and is in demand for this purpose by farmers. The two substances together can be worked into a species of cellulose, an operation which is only found profitable when conducted in boilers specially constructed for the purpose. Chance pieces of bark, larger pieces of wood, the waste from the grinding apparatus, and the dust from the sieves cannot profitably be separately treated.

The last waste product to be mentioned, but the first in the actual manufacture, is the rag dust, which by itself constitutes an excellent fertiliser. The fibre it contains may be regained by stirring with water and running off the liquid after a short time, when the fibre is carried away, but the dust, sand, and all heavier particles remain behind.

The damaged paper is also to be reckoned as a by-product. It is unnecessary to say anything concerning its utilisation; it is always produced in every process; it is only mentioned as an example to demonstrate that the further treatment of waste matters is always profitable, and occasionally very profitable. Such utilisation, an important factor in the balance sheet of a paper or paper-pulp works, must increase in importance in view of the present low prices.

CHAPTER XXXV

BY-PRODUCTS IN THE TREATMENT OF COAL-TAR OILS

Utilisation of the By-Products.—The caustic soda solution used in refining oils contains creosote, which is partly used directly as soda-creosote for impregnating mine timbers, and is partly worked up into carbolic acid. For this purpose the liquors are mixed with the sulphuric acid, which has also been used for refining the oils, until the reaction is strongly acid. In this case, acid sodium sulphate is produced, which is more soluble, and less hinders the separation of the creosote than the more difficultly soluble neutral salt. The salt liquor is set aside to crystallise, and the separated salt sold to soda or glass-works.

In decomposing with carbonic acid, which, according to L. Grotowsky (*Zeits. f. Paraffin Ind.*; also see p. 186), is obtained from burning coal, the mixture of soda-creosote and water in equal parts must be cold. Carbonic acid is passed in until the liquid is saturated and froths up violently for about an hour. The separated soda solution is evaporated to dryness, ignited, dissolved in water, causticised with lime, the caustic liquor drawn off from the chalk mud, and concentrated to 35–38° B. The caustic soda solution thus obtained still contains impurities, and can only be used to separate the creosote from crude oils.

The creosote, separated by one or the other process, after washing with water, may be brought into the market; it is used for soaking telegraph poles, railway sleepers, etc., also as a disinfectant. When distilled, it gives a fluid oil of 0·965 specific gravity and with a strong odour, which is used as a disinfectant under the name of creosote oil, or in the manufacture of phenol dyes. By repeated solution in caustic soda,

decomposition with sulphuric acid, distillation over iron filings, and finally over green vitriol, a quite white oil is obtained. The substance produced under the name of creosote by the mineral oil works in Saxony is, according to Grotowsky, simply phenol and cresol.

The acid resin drawn off from the refining apparatus is mixed with hot water in suitable vessels, and washed by direct steam ; on standing the empyreumatic resin separates out at the top. The recovered acid, which is coloured black by organic matter, is sold at a strength of 40–50° B. to superphosphate works, where it is used for dissolving phosphates and bones. The resin is well washed, mixed with any unutilised creosote, and subjected to distillation, when, according as tar or pitch is required, little or much creosote oil is distilled off.

The bone black used in refining paraffin is boiled out with water, then heated in horizontal retorts to drive off the residual paraffin, and finally converted into superphosphate.

CHAPTER XXXVI

FUR WASTE

Utilisation of Furriers' Waste.—Furriers produce a quantity of waste, which can only partially be employed, by neat and careful joining, to produce fur articles. A large proportion of the smallest cuttings constitutes valuable material for the hatmaker. No furrier, when cutting, should neglect to have at his side a small basket, into which the clippings can be thrown; otherwise, after first being dirtied on the floor, they have to be sorted out from other cuttings of all kinds. The waste pieces may be utilised as described below.

The waste grey pieces of monkey fur are used to fill the star-shaped and round mosaic fur covers of opera-baskets.

The feet and heads of astrachan, carefully fitted together, afford a material for covering gloves and hunting-mittens.

Pieces of bear-skin of all kinds are in great demand by brushmakers.

Cuttings of beaver and musk, even the smallest, are sought after by hatmakers at a good price. The forehead pieces of beaver-skins, placed together with the hairy side outwards, may be well used as trimmings on furs; larger pieces are very suitable for caps. The heads of musk, with the eyes and ears taken out by two cuts going from the eye over the ear, cut into triangular shape and joined together starwise, may be used for linings, as also may the short ends.

Long-haired pieces of badger, such, for example, as are cut out of the middle in making covers for knapsacks, are highly prized by brushmakers.

Fox: the tails are used in making boas, and are also the most valuable material for ornamenting opera-baskets, footwarmers, gloves, etc.

Goose and swan: the feathers are plucked from the waste

pieces and used for stuffing muffs. Powder-puffs may be made from the larger side pieces.

Grebe: wings joined to heads, or half small wings, make handsome ornaments for bonnets of seal and rabbit-skin, or of seal and musk.

Hare: the ears of hares are frequently set together, *i.e.* stitched near together on linen, and used in making hunting-mittens and caps. This process is, however, not to be recommended. The hatmaker takes all the rest, with the exception of the black pieces, which may occasionally be sold to toy-makers.

Ermine scraps are frequently bought in order to make imitation tips; but this is only done when the pieces are fine and of good size, and ermine skins are very dear.

Polecat: the scraps are bought by the Greeks to make linings; the heads, when preserved, on account of the expression, are used for ornamenting hunting-muffs; also, when cut into triangular pieces, for the mosaic covers of opera-baskets. The tails are used in brushmaking.

Rabbit-skins: the heads of large skins are put together to make wristbands, those of small skins for trimmings. Large sides may often be put together to make collars. All the rest, with the exception of the black portions, is used by the hatmakers.

Cat-skins produce little waste. Good cat-skins when damaged may be used as tips in white rabbit muffs. Tails are practically of no value.

Lynx: the feet may be made into cheap trimmings. There is practically no other waste.

Marten: the foreheads may be used for the mosaic covers of opera-baskets, as also the throats, which may be used together with the feet for linings similar to sable, but heavier. The tails are used for decorating ornaments, and also in brushmaking.

Mink: the cuttings are put together by the Greeks; the tails, like those of the marten, are used for ornamenting ladies' trimmings, and for borders. The foreheads are also used for making the fur covers of opera-baskets.

Otter: fine material for hatmaking, but generally put

together by the furrier, plucked, and dyed. The tails, when put together, form handsome and durable hunting-muffs.

Sea-otter: good pieces are highly prized; in Russia they are principally joined together for cap linings.

Sheep-skins: large pieces are joined together for lining opera-baskets.

Raccoon: the hairy sides are joined together for sleeves, or even for fur linings; the heads are used for trimmings on black fur with raccoon borders; the tails give durable trimmings.

Sealskin pieces are valuable. In England the smallest are joined together for caps, etc.

Skunk: the tails are bought by brushmakers. The white and black foreheads, properly joined together, and often worked in with triangular pieces of black cat-skin, make very handsome trimmings, especially for fox-skin.

Goat-skin waste is bought by brushmakers.

The Waste of Tanned Sheep and Lamb-Skins, as produced in the clothing industry, is mainly utilised by shaving off the wool, which is used in cloth-making, whilst the leather is bought by makers of prussiate of potash, hide-glue, and artificial manures. Gawalowski has discovered a method which makes it possible not only to separate the wool from the leather in its original length and without loss of strength for spinning and textile purposes, but also, when possible, to obtain the leather in a commercially useful form.

If the skins are immersed in a moderately strong solution of caustic potash or soda, the wool hairs are in a short time entirely dissolved, and there remains a bare, slippery hide, which, after well washing with water, is an excellent raw material for the manufacture of prussiate of potash or glue. Since the wool is entirely dissolved in the alkaline solution, the latter can be used for the preparation of ammonia, and especially for "animalising" vegetable fabrics, so that they may dye more rapidly. This process may be applied in the manufacture of jute carpets, and in weaving jute tapestries and hangings.

If the skins are immersed in strong ammonia, the leather is attacked in such a manner that, on taking out after an

immersion of some duration and drying, first in the air, then at 30–40° C., it is now in the form of somewhat friable pieces of skin. It is then only necessary to bring the wool into a tearing or beating machine, and next to card it, in order to obtain, on the one hand, the wool entirely as a good textile material, and, on the other, the leather waste in the form of a hide powder, which, like flesh-meal and bone-meal, is an excellent fertiliser.

If the skins are immersed in a moderately strong solution of an alkaline carbonate—carbonate of potash, etc.—the hide swells up considerably in two to five days, when the wool can be easily and completely separated by suitable apparatus from the moist and pliable skin, so that, on the one hand, there results a leather waste, as in the first method, and, on the other, wool exactly as in the second method. This last process, accordingly, unites the advantages of the former methods, it is the most simple and most lucrative, and also, up to the stage of the mechanical separation of the wool, the cheapest. Prussiate of potash works, which work up "dry liquor" into black potash, may combine this minor industry with their main process in a rational and lucrative manner, since the leather is an excellent material for adding to the melt, and their black potash is already obtained containing nitrogen. The saleable wool will then produce a not inconsiderable profit.

CHAPTER XXXVII

THE WASTE PRODUCED IN THE MANUFACTURE OF PARCHMENT PAPER

Utilisation of Parchment Paper Waste.—C. D. Cech has given an account of the utilisation of the waste from parchment paper works in the preparation of oxalic acid. He remarks that the chief attention in making oxalic acid from parchment paper waste must be devoted to a thorough washing of the latter. According to the method of manufacturing oxalic acid introduced by Roberts, Dale, & Co., of Warrington, by fusing sawdust with caustic potash (see p. 110), parchment paper waste must not only give a sufficient yield of oxalic acid, but its preparation from this material would not be affected by the colouring matters which arise in the treatment of hard woods.

The process of manufacture is the same as in the preparation of oxalic acid from sawdust (compare the details of this application of sawdust). J. Upmann (Post's *Zeits. f. d., chem. Grossgew.*, II. 4) remarks, in regard to Cech's proposal, that the process of converting cellulose into oxalic acid by fusion with alkali is not new, since it had already been confirmed by experiments on the small scale, before the introduction of the present usual method of making oxalic acid; on the other hand, it cannot be denied that there had been no previous published proposal to utilise cellulose in this manner. Now, Upmann considers that whether parchment paper waste may really be a suitable substance to replace sawdust cannot be decided *a priori*, since, apart from the question whether sufficient raw material could be obtained, it would have to be compressed for transport to a distance: and the complete washing would be attended with more difficulty than would be believed at first sight. Also, the subsequent drying of the paper could not well be obviated; thus there would be an expense which is lacking in the case of sawdust, but which might be compensated by a larger yield of oxalic acid.

CHAPTER XXXVIII

MOTHER-OF-PEARL WASTE

Utilisation of the Waste of Button-Making.—A substance, which formerly could not be utilised, is the lining cut out by button-makers from the shells, the innermost layer of which is the so-called "mother-of-pearl." The suggestion was made to turn this waste into a fine powder, when a soft bronze powder, of a silky, metallic lustre, was obtained, which can be employed in many ways, especially since it can be coloured to any shade. The pulverisation of the mother-of-pearl waste is accomplished in the same manner as that of wood which is to be worked up into wood-pulp; *i.e.* the waste is ground by a suitable rotating grindstone, in such a manner that it is first beaten to pieces, and then brought into a cylinder, in which it is pressed on the grindstone by means of a piston. The powdered mother-of-pearl obtained in this manner is then levigated, and, after drying, brought on the market, with or without previous dyeing. In order to convert mother-of-pearl into a glittering powder, suitable for makers of artificial flowers, the shells are first treated in closed vessels with high pressure steam, after which they can readily be powdered. The use of this powder in making wall papers is also said to be profitable: for 1 sq. metre there is required 3·5 grms.; thus, for a roll of paper 9·5 × 0·5 metre (31 ft. × 20 in.) there would be required about 14 grms. ($\frac{1}{2}$ oz.) of powdered mother-of-pearl, the cost of which is 1 pfennig per grm., or 10 marks per kilo. (4s. 5d. per lb.). The chemical works of M. Hess, at Neufünfhaus, Vienna, produce "pearl silver."

CHAPTER XXXIX

PETROLEUM RESIDUES

Preparation of a Resin-like Substance from the Sulphuric Acid used in Refining Petroleum.—W. P. Jenny, of Boston, has obtained a patent for the preparation of a substance similar to resin from the waste sulphuric acid of petroleum-refining. The sulphuric acid, which has been used for refining petroleum or shale oil, is mixed with an equal volume of water, when two layers form; the upper consists of a thick oil of objectionable odour, the lower of dilute sulphuric acid. The oil is repeatedly washed with boiling water, finally with the addition of a little soda, and is then distilled. After the oil distilling below 250° C. has come over, a current of air is blown by means of a suitable arrangement for forty-eight hours through the residual oil, the operation being conducted in the retort used for the distillation. During this process the oxygen of the air is eagerly absorbed by the oil, which, on cooling, solidifies to a deep brown mass similar to resin.

Another method for obtaining this resin consists in heating the residual acid, which contains the oil in solution, for several days at 100–150° C., until a test portion sinks when poured into water. The mass is then poured into water and washed with a large quantity of water, or, if necessary, an equal volume of petroleum ether is first added. In the former case the resin separates on the bottom of the washing vessel; in the latter case it remains dissolved in the naphtha, and floats on the surface of the acid.

The product, which is of a more or less hard nature according to the extent of the oxidation, is insoluble in water, alkalis, and alcohol, but dissolves readily in all fats, animal and vegetable oils, naphtha, benzol, chloroform, ether, carbon

bisulphide, amyl alcohol, spirits of turpentine, and in sulphuric acid of 66° B. When melted with gutta-percha or caoutchouc in varying proportions, it forms an elastic substance which can be used for insulating purposes.

Tar and other Products from Petroleum Residues.—Letny has investigated the composition of the tar obtained in making illuminating gas from petroleum residues (*Polyt. Journ.*, 229, p. 353; Post's *Zeits. f. d. chem. Grossgew.*, III. 30, 244, 506). In this method of making gas the petroleum residues are subjected to dry distillation in retorts filled with wood, when they produce illuminating gas and a tar of specific gravity 1·015. If the latter is taken through the retorts a second time, only a very small quantity of gas is produced, and the tar is but little altered; it has now the specific gravity of 1·207, and possesses all the properties of coal-tar. It begins to distil at 80° C., and gives fluid distillates below 200° C.; at higher temperature solid substances are formed, but later, above 300° C., oils again come over, which, however, separate crystalline compounds. A more exact examination of the distillate gave—below 200° C., 13·9 per cent., containing 4·6 per cent. of benzene, 5·2 per cent. of toluene and xylene; from 200–270° C., 26·9 per cent.; from 270–340° C., 8·6 per cent., containing naphthalene, cumene, and petroleum; above 340° C., 27·5 per cent., containing 3·1 per cent. of pure anthracene; the residue in the retort was 20·6 per cent.

This tar affords the possibility of utilising the petroleum residues in districts, which are rich in petroleum, to produce substances which can be used in the manufacture of aniline dyes and alizarine. The quantity of petroleum residues produced in Russia alone was estimated by Letny at 98,000 tons in the year 1878. Of this only a small portion was used for heating the distilling apparatus, another portion went into the interior of Russia for gas-making, whilst more than half remained, and was burnt up outside the works, owing to lack of storage. There is no doubt that the future will find in these petroleum residues a valuable material for the production of aniline, alizarine, etc.

In order to ascertain the conditions most favourable to

the formation of tar, petroleum residue of specific gravity 0·872, and boiling at 270° C., was passed through red-hot iron pipes 1·24 metres (4 ft. 1 in.) long and 5 cms. (2 in.) wide, filled with carbon. Irregular heating gives rise to very different results. In one case 100 grms. gave 23·9 grms. of tar and 30·1 litres of gas; in another experiment 46·4 grms. of tar and 59 litres of gas. At higher temperatures the yield of gas was higher—up to a certain temperature, above which the yield again became less. Without carbon the decomposition in the pipes proceeded irregularly, until carbonaceous deposits had formed from the petroleum decomposed, which deposits then facilitated the decomposition. The action is the same when the pipe is filled with broken brick, asbestos, etc. Very porous carbon, platinised, was still better than ordinary carbon; those substances which most readily absorb gases cause the production of the most tar. If the tar produced be again passed through the pipe under pressure it is considerably decomposed, carbon being deposited. Without increase of pressure, however, it suffers practically no alteration. The specific gravity of the tar never exceeds 0·8889; it contains a mixture of amylene and benzene, 6·28 per cent.; benzene, 12·56 per cent.; toluene, 10·5 per cent.; mixture of toluene and xylene, 6·8 per cent.,—in all, 52·5 per cent. boiling below 320° C., and 35·0 per cent. above 320° C. Solid hydrocarbons, such as naphthalene and anthracene, could not be detected, which is ascribed by Letny to the fact that the petroleum vapours have passed through too short a layer of carbon. In fact, when gas retorts are used, the yield of tar is lower the less coal is present in the retorts; accordingly, the quantitative yield of tar, and the percentage of solid hydrocarbons in it, is proportional to the dimensions of the layer of carbon through which the petroleum vapour is passed. In working on the small scale, amylene, hexylene, etc., were always formed; but not when gas retorts were used. Steam passed through the retort together with the petroleum residues was without action; and, on the other hand, by the application of pressure the yield of tar was always reduced. Similarly to the tar, the gas differed in composition according as it was made

on the large or small scale. In the latter case it contained, in addition to hydrogen and marsh gas, ethylene, propylene, butylene, acetylene, and crotonylene.

Petroleum Residues as Fuel for Cupola Furnaces.—The introduction of pulverised fuel in cupola furnace practice effects, on the one hand, the oxidising action of the excess of blast, and, on the other hand, prevents the formation of protuberances on the tuyères, and the attendant inconveniences. The carbon deposited on the walls of gas-retorts, and the residues obtained in petroleum refining, are the most suitable fuels to be used in the pulverised state, since they are completely free from ash. W. Batty, of New York, injects these substances, in a state of fine dust, with the blast, and obtains the following advantages:—(1) The whole of the oxygen, as it enters the zone of fusion, can at once combine with carbon, by which means a powerful neutral or reducing flame is produced;—the quantity of carbon introduced can readily be regulated as may be necessary; (2) the iron becomes hotter, and instead of oxidation occurring through the presence of an excess of air, the iron readily takes up carbon in the atmosphere of carbonic oxide impregnated with heated carbon; (3) in consequence of the higher temperature, the slag is more fluid; hence (4) the formation of protuberances on the tuyères is avoided; (5) the loss of metal is lower. On account of the partial carbonisation of the iron, the possibility of selecting scrap-iron, which is, as far as possible, free from sulphur and phosphorus, is of especial importance to the quality of the castings.

Utilisation of the By-Products and Chemicals of Mineral Oil Works.—Grotowsky has made certain noteworthy observations on this subject (Post's *Zeits. f. d. chem. Grossgew.*, III. 2, p. 345), according to which the carbolic acid may be separated from the so-called creosote-soda, produced in refining the crude oils, either by means of the dilute sulphuric acid, also produced in refining, or by means of carbonic acid made for the purpose. In the former case Glauber's salt, in the latter soda, or, in the event of again causticising by lime, caustic soda, are produced. The separated carbolic acid is either merely washed with water

and then brought into the market to be used for disinfecting purposes and for saturating wood, or it is converted by distillation into the so-called creosote oil. The latter is then used for making phenol dyes or for disinfection. By repeated solution in caustic soda, decomposition by sulphuric acid, distillation over iron filings, and finally over green vitriol, a quite pure pale oil is obtained. The empyreumatic resin, which dissolves in the sulphuric acid during refining, is separated from the acid again by high-pressure steam. The dilute sulphuric acid then obtained may be applied in the manufacture of superphosphate, or as indicated above. The separated empyreumatic resin is washed with water and subjected to distillation either alone or together with creosote. In the distillation more or less is removed according to the purpose for which the product is intended: in the case of tar for roofing felt, only until the residue forms a stiff paste, but for asphalt, to be used as the binding medium for coal briquettes, until the mass, when cold, has a shining, brittle fracture.

CHAPTER XL

PLATINUM RESIDUES

1. The Residue which remains after Treating the Crude Ore with Aqua Regia.—In a clay crucible with a thick bottom, 1 part of the platinum residue (300–400 grms. at the most in one operation) is fused with an equal weight of granulated lead and 1·5 part of litharge, or with 1 part of granulated lead and 2 parts of a flux, consisting of 1 part of fluorspar and 1·7 part of anhydrous gypsum. The mixture is stirred with a pipe stem, the crucible broken and allowed to cool, when the lead regulus is carefully freed from the slag and dissolved in hot, fairly strong nitric acid; the platinum metals which remain undissolved are treated as described under (*c*). Since a little palladium has dissolved with the lead, the latter is precipitated by sulphuric acid, the solution evaporated to dryness, the residue dissolved in water, filtered, and the palladium precipitated from the filtrate by mercuric cyanide

2. The Residue of Impure Ammonium Chloroplatinate obtained on concentrating the liquid, from which the greater part of the platinum has been precipitated, is essentially the chloroplatinate.

3. The Solution Filtered off from the Impure Ammonium Chloroplatinate, which is either evaporated to dryness and the residue ignited, or from which the metal is precipitated by iron or zinc, is especially rich in rhodium.

Wöhler has recommended the following method for the separation of the metals:—

(*a*) *The impure ammonium chloroplatinate* (2) is reduced by ignition, the metals dissolved in *aqua regia*, the solution evaporated almost to dryness, taken up in a little water, mixed with twice its volume of spirits of wine and a saturated

solution of potassium chloride added. The precipitate consists of the potassium double chlorides of platinum and iridium, with traces of rhodium and palladium. It is filtered off and washed with alcohol containing potassium chloride. After drying, it is brought into a porcelain crucible, where it is well mixed with its own weight of sodium carbonate. The filter is burnt, and the ash added to the mixture. The mass is then gently heated until it becomes black throughout. The platinum is thereby reduced, but palladium, iridium, and rhodium oxidised. The mass is extracted with water, the potash withdrawn from the oxides by means of hydrochloric acid, and the whole washed, dried, and ignited; it is next, in order to separate the rhodium, fused with six parts by weight of potassium bisulphate at a gentle and long-continued heat. The rhodium salt which is formed is extracted with water, and the yellow solution boiled with hydrochloric acid, when it becomes red owing to the formation of the chloride. Caustic potash precipitates from this solution brownish-red rhodium hydroxide, which is reduced to metal by ignition in a current of hydrogen. The residue of platinum, palladium oxide, and iridium oxide is reduced by heating in a current of hydrogen, and, after cooling, treated with nitric acid, which dissolves the palladium. The liquid is neutralised by sodium carbonate, and a solution of mercuric cyanide added, when palladium cyanide is precipitated, which, after washing and drying, only requires ignition to be converted into metal.

A mixture of metallic platinum and iridium oxide remains. It is digested with dilute *aqua regia*, which extracts a portion of the platinum, the clear solution is poured off, saturated with ammonia, evaporated to dryness, and carefully ignited, when pure platinum remains. The residual iridium oxide containing platinum is evaporated to dryness with sodium chloride and *aqua regia*; the platinum salt is extracted from the residue by water, whilst the iridium oxide remains undissolved. The latter is filtered off, washed first with common salt solution, then with ammonium chloride, dried, ignited, and reduced to metal by heating in a stream of hydrogen. The filtered solution contains a small quantity of iridium, which is separated by evaporating to

dryness, heating the residue, as above, with sodium carbonate, and extracting the platinum from the washed residue with dilute *aqua regia.*

(*b*) *The Liquid Filtered from the Impure Ammonium Chloroplatinate, or the Metals Precipitated therefrom by Iron or Zinc.*—The metallic powder is dissolved in *aqua regia,* and then treated in the same manner as the filtered liquid, which is evaporated to dryness, the residue digested with strong *aqua regia,* potassium chloride added, and the solution evaporated to dryness on the water-bath. Ferric and copper chlorides are then extracted by alcohol, the residual double chlorides of rhodium, palladium, and iridium are fused with potassium bisulphate, and the mass treated with water, when iridium oxide remains. The liquid contains palladium and rhodium; the former may be precipitated by mercuric cyanide, and then the latter with potash, after boiling with hydrochloric acid, as in (*a*).

(*c*) *The Residue Insoluble in Aqua Regia.*—The black levigated mass is intimately mixed with about an equal volume of ignited and finely ground sodium chloride, and the mixture heated in a porcelain or glass tube in a slow current of moist chlorine, until the gas begins to pass through unabsorbed. The further end of the tube opens into a well-cooled, tubulated receiver, from the tubulure of which the excess of chlorine is passed into alcohol. The double chlorides of sodium with iridium and osmium are produced; the latter is mainly decomposed by the moisture in the chlorine, the resulting osmic acid sublimes into the receiver, and is partially carried into the alcohol. After cooling, the residue in the tube is treated with water, and then washed with hot water. The dark reddish-yellow iridium solution, filtered off from the ferric oxide, is mixed with strong nitric acid and distilled, when more aqueous osmic acid passes over. When the liquid has in this manner become very concentrated, it is mixed hot with a strong solution of ammonium chloride. On cooling, a large portion of the iridium separates as the crystalline blackish-red ammonium iridium chloride, which is filtered off and several times washed with ammonium chloride solution. On ignition it yields a grey sponge of iridium. A

small quantity of platinum in this salt is detected by grinding it very fine, mixing with eight times its quantity of water and leading in sulphur dioxide, when the iridium salt dissolves, leaving the yellow platinum salt. The residual solution is mixed with soda crystals in excess, evaporated to dryness, the mass gently ignited in a crucible, and, after cooling, extracted with hot water, which generally becomes coloured yellow by alkaline chromate. The black powder which is left consists of a compound of iridium sesquioxide with soda, contaminated with ferric oxide. It is reduced by gentle heating in a current of hydrogen, after which water extracts caustic soda, and the iron is removed by digestion with stong hydrochloric acid. If the residue be then digested with very dilute *aqua regia*, a little platinum is generally extracted, and may be precipitated by ammonium chloride.

In order to extract ruthenium, the iridium must then be fused for at least an hour in a silver crucible with a mixture of potassium chlorate and caustic potash. The mass is treated with water, the yellow solution of potassium ruthenate, which has been allowed to clear by settling (not filtering), is poured off, the residual iridium oxide repeatedly washed by decantation, and the solution of potassium ruthenate neutralised by nitric acid, when black ruthenium sesquioxide is precipitated. The latter is reduced to metal, in the same manner as the iridium oxide, by heating in a current of hydrogen.

The iridium, after this purification, is strongly compressed and subjected in a crucible to the fiercest white heat, when it is obtained in a somewhat coherent and dense condition.

The platinum residue, after one such treatment, is not exhausted; further quantities of osmium and iridium can be obtained by repeating the same operations.

CHAPTER XLI

BROKEN PORCELAIN, EARTHENWARE, AND GLASS

Broken Porcelain and Pottery with White or Coloured Glaze.—In the journal of this industry it is proposed to use these fragments for mosaic pavements for entrance halls, foot-paths, etc. The method is particularly suitable for roofing felt. The fragments are sorted according to colour, coarsely ground and then sieved through sieves of more or less coarse mesh. The mosaic pattern, drawn or stencilled on strong paper, is now covered or pasted over with the fragments according to size, the flat side downwards; the paper is surrounded by a rim and fluid asphalt poured on to a depth of two inches. After cooling, the paper is washed off, when the plate is finished. Large surfaces of pavements are made from separate plates, the joints being filled with melted asphalt. For this purpose pure asphaltum is not required; it is mixed with coarse sand, powdered asphalt stone, clinker, etc., and with a little lime.

The Waste from the Manufacture of Fine Earthenware.—This is a case of utilising the used gypsum moulds (*Zeits. f. Thonwaaren-Industrie*). It is recommended to employ these moulds for fertilising purposes, which has been found practicable. The author of the communication says:—"The main water trenches are made somewhat wider and deeper than usual and piled up with a layer of old gypsum moulds, in which the water finds sufficient space to penetrate. As soon as the moulds are dissolved, which is the case in a relatively short time, the layer is renewed. With this method of fertilising, the grass stands straighter and higher and shows more bluish-green colour, the clover between grows rapidly, the moss in wood-meadows visibly

disappears, and the crop is larger than in meadows not watered with the gypsum solution."

Utilisation of Broken Glass.—Postwing, Gatchey, and Geille, of Paris, have proposed to use broken glass in the manufacture of pilasters (*Thonindustrie-Zeitung*, 1891; *Neueste Erfindungen und Erfahrungen*, 1891). A quantity of broken glass of different colours, previously broken up to a determined size, is mixed together, brought into moulds lined with a resistant material, such as silica or talc, and heated. A coherent mass is obtained, which can be dressed and cut into blocks of irregular colour. These blocks may be used as artificial marble; they are generally rough on one side, and occasionally incompletely fused, thus giving surfaces which can readily be built up with the aid of a little mortar. Handsome decorative effects may be produced whilst the blocks are still plastic. When a mould is used which can be taken apart, the pieces of glass can be so divided off that very effective coloured glass windows are obtained during the heating, in which windows the necessity of the usual lead work is avoided.

Recovery of the Waste Gold in Porcelain or Glass-works.—All broken or spoiled porcelain or glass articles which have been gilded must be carefully kept. The fragments must be broken into small pieces and placed in a shallow vessel of suitable size. Then sufficient *aqua regia*, made from four parts of hydrochloric acid and two parts of nitric acid, is poured on to cover the fragments completely. The vessel is covered with another similar one of an equal diameter at the top, and the edges in contact carefully smeared with clay to prevent the vapours from escaping. After two to three hours it is advisable to ascertain whether the acid has exerted its action. If this is not the case after this period, the vessel is placed in another filled with boiling water, but generally a good result is obtained in two hours. The gold waste is freed from all acid by frequent washing with pure water. When the fragments are taken out of the acid, the gold is found at the bottom of the vessel. The oil and turpentine dishes are first freed from spirit, etc., by subjecting them, in an iron vessel, to the fire; they are

then treated in the same manner as the broken glass and porcelain. Cloths containing gold are collected and burnt to powder in an iron vessel. In the English porcelain and glass-works the waste gold is recovered in this manner, and the process has been found satisfactory.

CHAPTER XLII

SALT WASTE

Treatment of the Stassfurt Waste Salts.—Whilst referring here to one of the best and most comprehensive publications, *Die Industrie von Stassfurt und Leopoldshall und die dortigen Bergwerke in chemisch-technischer und mineralogischer Hinsicht betrachtet*, by G. Krause (Cöthen, with twenty-two wood-cuts), we shall follow in general the description of S. Pick (*Die Alkalien*, 2nd ed., Vienna, Hartleben) in regard to the treatment of carnallite, and draw therefrom certain further conclusions.

Carnallite is treated by a process which involves the separation of its constituents by crystallisation. From the crude salt a concentrated solution of potassium chloride is prepared by the introduction of steam. The solution then comes into clearing basins, from which it flows to the crystallising vessels. The residue is again boiled with water, and the resulting weak solution used in the next operation in place of water. The extracted residue is calcined, and its proportion of potassium chloride increased to 15–18 per cent. by the addition of potassium chloride or other waste matters containing potash, when it is finely ground and brought into the market as a fertiliser under the name of "prepared kainite." The residue may also be dissolved and exposed to the frost, when Glauber's salt is obtained, or it is used in the preparation of kieserite, by treating with a stream of cold water, which dissolves the smaller lumps of rock salt, leaving the larger, and carrying the kieserite away. A channel leads the water on to a sieve, where only the fine kieserite penetrates, whilst the larger pieces of foreign matter remain behind. As soon as the kieserite has collected in the settling tanks, it is shovelled out into

moulds, in which it soon takes up water and sets to solid blocks.

The hot, strong solution of carnallite, after settling, may be allowed to cool to 60–70° C. before the real crystallisation, when a large part of the sodium chloride crystallises out with a small quantity of potassium chloride. Or the solution is at once brought into high crystallising vessels; the potassium chloride, which crystallises out, still contains sodium chloride, magnesium chloride, and very small quantities of magnesium sulphate. These impurities are removed by placing the salt in wood or iron vessels with perforated false bottoms, pouring water over, allowing to rest for some time, and running off the resulting potassium chloride solution, which is used to dissolve carnallite. As a rule, another similar treatment follows, when a salt of 90 per cent. strength is obtained. The potassium chloride is then freed from the adherent moisture, either by calcination in kilns heated by the waste heat from pans, or on iron plates heated by steam.

The mother liquor left after the crystallisation of the potassium chloride, which still contains considerable quantities of that salt, is concentrated in evaporating pans. The salt which separates during the evaporation is generally calcined and added to the manurial salts. Formerly the liquor was several times evaporated to a small extent and left to crystallise after each evaporation. Now, in consequence of the fall in prices, it is only evaporated once, but as far as can be done without much injury to the pan—as a rule, to 35° B. From the clarified liquor artificial carnallite separates on cooling; the residual mother liquor is either run to waste or worked up for bromine, or it is evaporated to 40° B. and run into barrels, in which the magnesium chloride solidifies.

The artificial carnallite affords a source of very pure potassium chloride. It is dissolved in hot water to form a solution of 31° B., from which coarse potassium chloride crystallises on cooling. The crystals are treated in the same manner as the first crystallisation from the crude potash salts, when a very pure product results.

The second and more recent method for treating natural carnallite is founded on the fact that hot magnesium chloride

solution takes up the total content of the natural impure carnallite and deposits it again in the crystalline state on cooling, in a very pure condition, so that, on decomposition with water, it produces very pure potassium chloride. The mother liquor is again applied to the solution of fresh quantities. The mother liquors resulting from the decomposition of the artificial carnallite are evaporated, when they deposit small quantities of "stage salt" and give a fresh crop of carnallite. This process offers great advantages: pure potassium chloride is obtained, and there is less liquor to evaporate; but, on the other hand, it requires complicated machinery.

The residue obtained in working up the saline deposits for calcium chloride, amounting to about 30 per cent. of the crude salts, is sold to green glass-works under the name of "glass-melt." It is there well dried, finely ground under edge runners or in a roller mill, and is then best at once mixed with the other substances used in the charge. The air-dried residue contains: potassium chloride, 5·25 per cent.; sodium chloride, 47·0 per cent.; magnesium chloride, 3·85 per cent.; magnesium sulphate, 29·25 per cent.; insoluble matter, 8·15 per cent.; water, 6·50 per cent.

G. Borsche, of Leopoldshall, near Stassfurt, has patented a process for the preparation of potassium sulphate from kieserite and potassium chloride. A mixture of 5–8 equivalents of potassium chloride and 4 equivalents (2 molecules) of magnesium sulphate is treated in succession with small quantities of cold or moderately warm water, insufficient to dissolve the salts completely. For example, 6 equivalents = 447 grms. KCl, and 4 equivalents = 552 grms. $MgSO_4 . H_2O$, are treated four times in succession, each time with 300 grms. of water, after each digestion the liquor being allowed to drain away. The residue is then almost pure potassium sulphate, and there are in all about 1200 grms. of water to evaporate.

CHAPTER XLIII

SLATE WASTE

Utilisation of Waste Slate.—At the slate quarries in Wales many million hundredweights of waste slate have accumulated; it is used as a raw material for brick-making. The slate in question is nothing but highly compressed clay, in which condition it has lost the property of taking up water, and hence is no longer plastic. It can thus only be worked into bricks by dry or semi-dry presses, and the bricks must be burnt until they sinter, in order that they may acquire solidity. The waste is now treated by machines specially constructed for the purpose. It produces bricks which are unsurpassed in denseness, strength, and hardness, whilst they are about 33 per cent. cheaper to make than ordinary bricks. They will also bear a very high temperature. Their crushing weight is given at 1135 kilos. per sq. cm. (16,500 lb. per sq. in.).

The method of making these clay bricks is as follows:—The clay waste is first pulverised; the powder then falls through a perforated iron plate into a pit, from which it is raised by elevators to a higher floor, where it is mixed in the cylindrical mixer with a small quantity of water. The slightly moist material then goes to the hopper of a brick press—a dry or semi-dry press—in the moulds of which it is compressed by high pressure into bricks. The latter are taken direct from the press to the kiln and burnt.

CHAPTER XLIV

SULPHUR

Recovery of Sulphur from Sodium Sulphide.—At Stolberg, near Aix, the sulphur dioxide obtained by roasting zinc blende, etc., is cooled, and then absorbed by sodium sulphide, under the action of steam, when sodium thiosulphate and sulphur are formed, which are removed in barrels, whilst the furnace gases, freed from sulphur dioxide, pass up the chimney. From the mixture of sodium thiosulphate and sulphur the latter is distilled off, the soluble matter—sodium sulphide and sulphate—extracted from the residue, the Glauber's salt separated by crystallisation from the sodium sulphide, and the former again converted by ignition with coal into sodium sulphide, which may again be used to absorb sulphur dioxide.

Sulphur from Coal Gas.—In purifying coal gas by means of Laming's mixture, there is obtained a product containing as much as 40 per cent. of free sulphur, from which sulphur can be obtained by distillation over an open fire, or with superheated steam, or by extraction with heavy tar oils or carbon bisulphide.

Sulphur from Gypsum, barytes, alkaline sulphates, and barium sulphide. The author of the article "Sulphur" in Muspratt's *Chemie* remarks that these methods, in spite of the cheap raw materials, have not yet attained to economic results, on account of the difficulty of separating the sulphur. Sulphuric acid has also become so cheap since its production on the large scale in smelting works, that it is used with advantage in making artificial gypsum for paper-works. (Also compare the process of Schaffner and Helbig, under "Recovery of Sulphur from Alkali Waste," p. 200.)

Sulphur obtained in the Manufacture of Iodine from Seaweed.—In making iodine in Scotland, France, and the Orkney Islands from kelp, the so-called vegetable sulphur separates from the mother liquors in large quantities, owing to the decompositions which take place.

Sulphur from Calcium or Potassium Sulphide is obtained as a precipitate (milk of sulphur), on decomposition by an acid (hydrochloric).

According to an English patent, J. Hollway, of London, obtains **sulphur from pyrites,** by passing steam over the pyrites at a dark-red heat, and heating to 800° C. About half the sulphur then distils over uncombined; the remainder is evolved as sulphuretted hydrogen, leaving a residue of ferric oxide.

Recovery of Sulphur from Alkali Waste.—Among the numerous methods for the recovery of sulphur, the majority of which can have no long existence since they have not succeeded in practice, the process of M. Schaffner and W. Helbig, of Aussig, which has been found practically successful, is to be regarded as especially good. These two chemists have succeeded in solving a long-attacked problem, by again making useful the plague of soda-works—waste black ash. Whilst by the processes hitherto used, which depend on the oxidation of the calcium sulphide, only half the sulphur has been regained, and all the lime, together with the other half of the sulphur, has remained as a second waste product, Schaffner and Helbig recover all the lime and all the sulphur in a usable form. Their process, as patented in Germany, for the *preparation of sulphur from soda residues, gypsum, barytes, and sulphurous acid, with the simultaneous recovery, as carbonates, of the earths united with the sulphur*, is based, in the first place, on the use of magnesium chloride to decompose the calcium sulphide, a reaction not hitherto applied on the large scale. The reaction is—

$$CaS + MgCl_2 + H_2O = CaCl_2 + MgO + H_2S.$$

The calcium carbonate is not attacked by the magnesium chloride.

The magnesium chloride employed is obtained by sub-

jecting to the action of carbon dioxide the residue, consisting of magnesia, calcium chloride, and the remaining unattacked constituents of the waste black ash, which remains from the action of magnesium chloride on calcium sulphide after the sulphuretted hydrogen has been driven off. Calcium carbonate and magnesium chloride are obtained according to the equation—

$$MgO + CaCl_2 + CO_2 = MgCl_2 + CaCO_3.$$

Instead of magnesium chloride alone, hydrochloric acid, to replace a portion, may be run in simultaneously or alternately. The acid at once dissolves the separated magnesia, when it can again exert its action. The sulphuretted hydrogen is converted into sulphur by means of sulphur dioxide, according to the equation—

$$2H_2S + SO_2 = 2H_2O + 3S.$$

However, not only sulphur and water are formed here, but also other compounds, such as tetrathionic acid—the inventors say pentathionic acid, which, according to the researches of Spring (Post, *Zeits. f. d. chem. Grossgew.*), does not exist; this reaction would not be applicable on the large scale if the inventors had not found a means of preventing the formation of tetrathionic acid, etc., or of precipitating the sulphur from it, if formed. This means consists in the use of a solution of calcium or magnesium chloride, in which the sulphuretted hydrogen and sulphur dioxide decompose one another. The theoretical quantity of sulphur is separated. If an excess of one gas or the other be present, it is without influence on the decomposition, and is found unaltered in the solution. The function of these chlorides in the reaction is not yet ascertained; it has, however, been found that about one equivalent of calcium or magnesium chloride is required to the total sulphur present.

Schaffner and Helbig have later discovered that the calcium and magnesium chlorides used in the main patent to prevent the formation of pentathionic acid, etc., in the conversion of sulphuretted hydrogen into sulphur by means of sulphur dioxide, may be replaced with equal success by hydrochloric acid, sodium sulphate, magnesium sulphate, barium

chloride, and sodium chloride. About one equivalent of these salts is used to the total sulphur present.

The alkali waste is decomposed in the warm by magnesium chloride in large closed iron tanks provided with stirrers. Either the black ash waste is gradually introduced into the total quantity of magnesium chloride required to fill the tank, or the magnesium chloride solution is run on to the total quantity of black ash waste, or, finally, the two are simultaneously and slowly introduced in equivalent quantities. Loss of sulphuretted hydrogen is avoided by preventing the accumulation of pressure in the tanks and decomposers, which easily is effected by flues, fans, or pumps introduced at suitable points. In the sulphuretted hydrogen decomposers there is also always maintained a larger quantity of sulphurous acid than is equivalent to the sulphuretted hydrogen coming from the developing tanks.

The siliceous and clayey matters (clinker) present in the black ash waste, which, if they remained in the regenerated lime, would soon accumulate to such an extent as to make it useless for soda-making, are removed by a process of stirring with water and allowing to settle, or by passing the decomposed residue through a fine sieve. The regeneration of the magnesium chloride and the lime from the decomposed residue freed from clinker is effected by the introduction of air containing carbon dioxide (fire gases, etc.).

The sulphuretted hydrogen coming from the tanks is mixed by means of mechanical contrivances in vats or towers with sulphurous acid and an aqueous solution of magnesium or calcium chloride. In the case of towers, the arrangement should be such that the sulphuretted hydrogen is introduced at the bottom and the sulphurous acid a little higher. The sulphurous acid required may be made from sulphide ores, sulphur, or sulphuretted hydrogen, or any furnace gases containing sulphide dioxide are taken and either conveyed direct to the decomposing apparatus or separately into a condenser, similar to those used for hydrochloric acid, where they produce a solution of sulphurous acid in water or in a solution of calcium or magnesium chloride.

This process may be applied as well to black ash waste as to gypsum and barytes after their previous reduction to calcium or barium sulphide. It may also be employed for utilising and removing the injurious sulphur dioxide produced in so many metallurgical processes.

To complete the account of this process, we add a private communication made by Schaffner to Dr. Post, the editor of the *Zeitschrift für das chemische Grossgewerbe*, according to which a little burnt dolomite—a double compound of magnesium and calcium carbonates—is added during the carbonation; this addition also yields magnesium chloride, and thus compensates for the unavoidable loss of this compound in working, amounting to 5–6 per cent. The treatment with carbonic acid, which has hitherto taken place in an apparatus similar to Weldon's manganese mud-oxidation tower by means of a powerful blowing-machine, is now more economically effected in an ordinary tower with currents of gas and liquid in opposite directions.

The position of this process in practice is best shown by an article on the subject in Post's *Zeitschrift für das chemische Grossgewerbe.* In answer to a question, whether the process of P. W. Hofmann for the regeneration of sulphur was applied anywhere except at Dieuze, Dr. Grüneberg stated that the Mond-Schaffner method was given the preference, *i.e.* that the precipitation was made by Mond's method and the fusion after Schaffner's method. Post states that the sulphur is now precipitated by Mond's method even in the works at Aussig, since Schaffner's precipitation apparatus, though working perfectly well, required too much labour and attention when working on an extensive scale. The oxidation of the waste is always effected by Schaffner's process, *i.e.* at first in heaps in the air, then by blowing in flue gases. (Mond blows air from a fan through the waste, adds an excess of the liquor to hydrochloric acid, then neutralises by hydrochloric acid, again adds sulphur liquor, and so continues until the vessel is full.)

K. Kraushaar has described in the *Polyt. Journ.*, a process used with great success for many years in the works at Thann for **recovering the sulphur in open vessels from alkali**

waste. According to Kraushaar's experiments, there forms in the interior of heaps, as far as possible protected from the entry of air, calcium hydrosulphide and hydroxide, water being absorbed. Calcium hyposulphite is not formed until oxidation occurs through air entering on breaking up the heaps, polysulphides being first formed, then hyposulphite. If now the loosened alkali waste be exposed to the air for just the proper length of time—determined by the examination of samples—that the liquors may contain to one molecule of calcium hyposulphite one molecule of calcium hydrosulphide and two molecules of calcium sulphide, CaS_2, then there is no evolution of sulphuretted hydrogen on the addition of hydrochloric acid, and consequently the decomposition may be performed in open vessels. At Thann, the process is continuous; acid and liquor in the proper proportions enter, near the bottom, a wooden vessel provided with a stirrer and heated by steam, whilst the decomposed liquor and the separated sulphur flow away continuously through a pipe near the upper edge of the tub. Kraushaar states that the process of forming the hydrosulphide is more rapidly effected than by stirring the heaps, by treating it with water and steam at five atmospheres pressure in sheet-iron cylinders; 90 per cent. of the sulphur in the waste can be recovered in this manner. The remainder is then made available by atmospheric oxidation. Kraushaar suggests the use of calcium hydrosulphide liquor for unhairing hides in tanning.

This process, Post remarks, was not found successful in small experiments in the works; 90 per cent. of the sulphur was not obtained. The calcium hydrosulphide appears to decompose again at the temperature employed, since on opening the apparatus streams of sulphuretted hydrogen are evolved. Since Kraushaar's process is much more costly than that generally employed, it could only count on adoption if it could recover considerably more sulphur than that hitherto obtained.

Mactear's utilisation of the yellow liquor from alkali waste.—According to Lunge (*Polyt. Journ.*), Mactear manufactured at Glasgow 1500 tons of sulphur per annum from

the yellow liquors which run from the enormous heaps of lixiviated black ash, and which had given rise to great complaint. The essential part of the process is the treatment of the liquor with an aqueous solution of sulphurous acid, either with or (better) without the addition of lime. Streams of the yellow liquor, of aqueous sulphurous acid, and of hydrochloric acid are allowed to run simultaneously into the precipitating vat, the volumes of the three solutions being so regulated that as little gas and as much sulphur as possible may be produced, just as Mond has described. A temperature of 70° C. is the most favourable for the reaction.

The lixiviated black ash can also find a further application in being used in place of lime in the **preparation of calcium pentasulphide.** If the waste be boiled with sulphur, the calcium sulphide takes up more sulphur and a solution is obtained which contains little hyposulphite.

CHAPTER XLV

BURNT PYRITES

Iron from Burnt Pyrites.—According to P. W. Hofmann (*Dingler's Journ.*, 215, p. 239) burnt pyrites is treated in the following manner in order to obtain the iron:—It is methodically extracted with water at about 40° C.; one equivalent of sodium chloride is added for each equivalent of sulphuric acid contained in the liquor, the Glauber's salt removed by cooling, and the mother liquor evaporated to 54° B. in order to obtain zinc chloride. The burnt pyrites is then removed from the washing-boxes, left for some days in the air to dry, and then sieved to separate the sulphurous material from that free from sulphur.

According to J. Cahen (*Ber. d. deutschen chem. Gesellsch.*), the burnt pyrites of vitriol works is well mixed with tar, turf, and limestone, the mixture made into lumps of suitable size, and treated in a blast-furnace. The iron obtained is said to be almost free from the ordinary impurities of pig-iron; any sulphur still present in the burnt pyrites combines with the lime, and the turf apparently effects the formation of volatile phosphuretted hydrogen.

CHAPTER XLVI

SILK WASTE

The Utilisation of Silk Waste.—The patented machine of Grothe and Heller remains the only one hitherto made for the manufacture of silk shoddy from silk rags, since it has been found that the quantity of silk rags is not great, and that the fabric cannot be entirely unravelled on account of the strength of the fibre. (Compare also the article on Shoddy, p. 251.) Broadly speaking, there are three branches of silk-waste spinning, which involve the manufacture of "floret" silk from first waste, noil yarn or "bourette" silk, and silk shoddy. The first raw material to be mentioned is the *strusa*, *i.e.* the clean waste obtained in unreeling the cocoons, from cocoons which cannot be reeled, and broken cocoon threads. A further distinction is made between *strusa à feli* and *strusa à vapeur*, according as it is obtained from cocoons which were unreeled from fire or steam-heated basins. *Strusa à vapeur* is accordingly the more valuable, and more in demand, to which also the fact contributes that it is produced in larger quantities of uniform quality, since it is made in the larger works. Perforated cocoons and whole cocoons which cannot be unreeled are not included in the *strusa*; these are brought into commerce under the term of "cocoons." *Strazza* is the name of the waste produced in spinning raw silk, through the breaking or spoiling of the threads. These materials also occur in a slightly decayed condition; the decay is said to improve the quality.

The second commercial raw material is decayed waste—*stami, peignées.* The *stami* are somewhat decayed cocoons. Marseilles is the principal market for silk waste; the following commercial qualities are sold there at prices rising from 1 franc to 16·5 francs per kilo. (4½d. to 6s. per lb.):—

Bassinets, Moorish, *frisons de nouca, de Khorassan,* Syrian, Accoulys (Greece), French, Italian, Persian, Broussa, Oshio, *Yama en Flottes, Kibisso Yama, Bourre de Chine,* Levant, Spanish, Adrianople, Salonica.

The damaged cocoons are brought into deep vats, hot water added, and the mixture allowed to stand for several days. The dead pupæ quickly cause putrefaction, which attacks and destroys the gummy and resinous matters. The material is then smoothed and cleaned by a combing machine, and brought into the market under the name of *peignée,* the fibres of which are from 5–16 cms. (2-6 in.) in length.

The flocks and unspinnable waste obtained in spinning the first waste have been utilised by Pasquay by spinning round hemp cords, and plaiting the cords, thus obtaining long bands, which can be used for wrapping steampipes to prevent loss of heat.

CHAPTER XLVII

SOAP-MAKERS' WASTE

Toilet-Soap Cuttings (*Neue Seifensieder-Zeitung*).—Manufacturers, who possess a pan heated by steam or in a water-bath, can readily melt together and again press the cuttings of toilet soap. The soap to be melted is cut up into shavings by means of a wire or plane, and brought, in small quantities at a time, into a pan heated by steam or on the water-bath. The soap is there allowed to melt, with the addition of a little water, or, if the soap becomes too thick and viscid, of spirit. The quantity of water to be used is determined by the condition of the soap; if it be dry more is added than if it were wet. If water-glass has been used in making the soap, it is necessary to add a few pounds of lye at 20–22° B. to prevent the separation of silicic acid. Naturally, care must be taken that the soap does not froth, which is accomplished by stirring diligently, and adding water if required. The water is only added in order to facilitate the melting of the soap, but not to dissolve it. When the soap is completely dissolved, the colouring matter is stirred in, and then the perfume, before pouring into the mould. These soaps are generally coloured yellow, red, or brown, and made into household, Windsor, mottled, coal-tar, and also sand and pumice soaps.

In melting the cuttings, if the soap should froth or become too thick and lumpy, and if a small addition of spirit remains without result, the following method is adopted:—Whilst stirring, sufficient water is poured into the pan to dissolve the soap completely to a thin liquor; it is then salted out with a strong hot solution of salt, the salt liquor removed, and the soap brought together by potash lye of 2–3° B. or hot water. After the soap has stood for some time, it is drawn off down to the deposit at the bottom, coloured,

crutched in the cold, perfumed, and poured into the mould.

The following directions for making brown Windsor soap by melting together cuttings may serve as an example of this method of treatment. For a brown soap are taken the cuttings of such coloured soaps as yellow, blue, and brown. Fifty lb. of soap are coloured brown by 120 grms. (4 oz.) of caramel, and then scented with 40 grms. of oil of lavender, 30 grms. of oil of thyme, 30 grms. of cumin oil, 30 grms. of oil of cloves, and 30 grms. of cinnamon oil. The caramel is obtained by carefully melting ordinary sugar in a pan, with

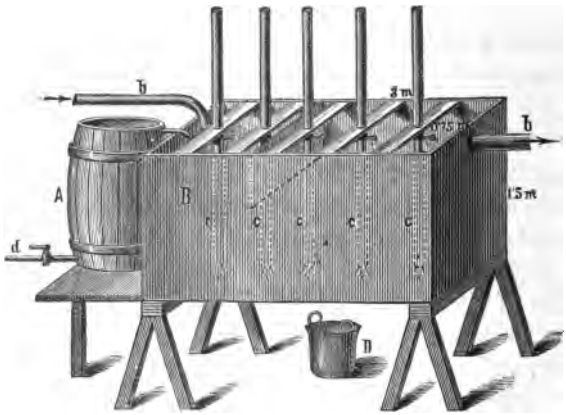

FIG. 10.

constant stirring, until it has changed into a mass, which runs from the stirrer in dark golden-yellow threads.

Utilisation of Waste Soapy Waters.—The utilisation of the waste soapy waters produced in such large quantities in public laundries and in many industries, *i.e.* the separation of the fatty acids which are combined with alkali in these waters, has been many times attempted, but with little success. The methods often recommended—collection of the soap-suds in tanks, decomposition of the soap, allowing to deposit, etc. (Heuze, *Dingl. Polyt. Journ.*)—can only be applied to small quantities of strong soap liquors, such as are produced in wool-washing. In the case of public laundries, which use

daily 150–200 kilos. (3–4 cwt.) of soap in 60,000–70,000 litres (13,200–15,400 gals.) of water, the application of such methods is quite impossible. In such cases, in order to collect the suds, the waste waters should be run, together with a regular stream of lime water, through the apparatus

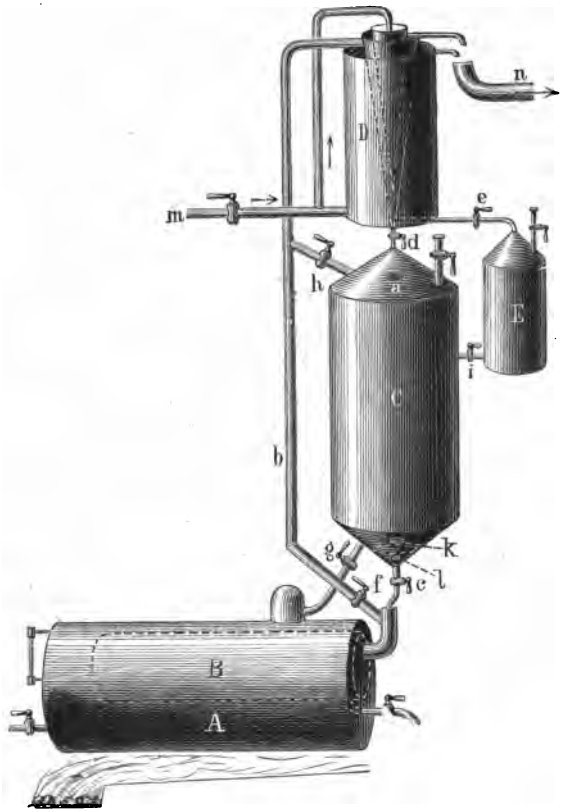

FIG. 11.

shown in Fig. 10. A tub *A* is half filled with slaked lime; a regulated current of water passes through the pipe *d*, the tub *A*, and the outlet, carrying lime into the vat *B*. The water from which the soap is to be removed enters at the same time in a continuous stream and leaves at *b*. It is then only necessary to run the mud deposited on the bottom of the

vessel into the filtering basket *D*, placed below, by raising the plugs *c*. The lime soap collected in the basket is decomposed with hydrochloric or sulphuric acid and steam, the cooled mass again brought into the filter basket, washed, and freed from water by draining and pressing. The fatty mass obtained is now by no means pure enough for the fat press. In the case of waters from a public laundry, it contains 40–50 per cent. of foreign matter—sand, pieces of wood, cellulose,—so that the production of fat therefrom is not a remunerative operation, apart from the cost of the apparatus. It is more economical to recover the fat by extraction with carbon bisulphide, a method already much in use. An apparatus very suitable for this purpose is shown in Fig. 11. It consists of the water-bath *A* and the boiler *B*, contained in *A*, for the reception of the fat. The impure fat is placed in *C*, *D* is the condenser and *E* the storage vessel for the carbon bisulphide. After the cylinder *C* has been filled through *a* with fat, which lies on a perforated bottom between the opening *k* and *l*, carbon bisulphide is admitted from *E* through the tap *i*. It dissolves the fat contained in the impure mass and carries it through *c* into the boiler *B*, in which the carbon bisulphide evaporates. The vapour rises through the pipe *b* into the condenser *D*, where it is condensed, the liquid then passing to *C*, whence it again carries fat to *B*. When all the fat has been removed in this manner, the taps *c*, *f*, *g*, and *h* are opened, whilst at the same time *e*, *d*, and *i* are closed. Steam then passes from *A* through *g* into *C*, expels through *h* the carbon bisulphide, which collects in *E*, together with that evaporated in *B*; *k* and *l* are openings for emptying the cylinder *C*. By this arrangement of the apparatus, not only is all the fat recovered, but also the loss of carbon bisulphide is reduced to a minimum, assuming that the necessary quantity of cold water is supplied to the vessel *D* through *m*. The fat remaining in *B* still requires refining, which is accomplished in the usual manner.

Utilisation of Liquids containing Fatty Acids.—In all the textile industries there is a number of waste liquors containing fatty acids, the quantity of which is so considerable that its recovery is at times a source of not inconsiderable

profit. In regard to the composition of these liquors, there are in general three different kinds to be distinguished: (1) The waste liquors obtained in removing the perspiration and grease from wool and woollen fabrics, to which are to be added the waste soapy waters of houses and large laundries; (2) the used soap liquors obtained in boiling silk to remove the "gum"; (3) the waste liquors of Turkey-red dyeing, which contain oil. In all the liquids mentioned the fatty acid compounds are mainly united to alkalis, accordingly their separation by a strong mineral acid appears to be suitable, and this method is almost always used when these liquids are treated. The method is, however, attended with so many inconveniences that many industrial establishments are disgusted with the utilisation of these waste products. In the first place the liquids, in addition to the alkali compounds, always contain lime soaps produced by the lime in the water. When the oils and fats are separated by sulphuric acid, or by crude hydrochloric acid, which always contains sulphuric acid, the lime is always separated as sulphate, which forms a pasty mass with the oil. This mass is not readily separated from the water, so that its application in making soap or illuminating gas is not easy. Also, the storage of the fluid oil in barrels is laborious and subject to considerable loss. Thus the separation of the fatty acids in the form of a solid compound of fairly constant composition appears to be indicated. Vohl has previously shown (*Musterzeitung*, 1873) that, when lime or a lime salt is added to these liquids, all the fatty matter is separated as a solid lime soap, from which the liquid can readily be drawn off, and which can be stored in the dry state. This lime soap is a substance which can readily be transported and sold to works occupied with the separation of oil and fat from such material. It is, of course, not the case that each establishment carries out the complete utilisation of its own soap waste, but simply that it removes the fat from its waste soap-suds with lime, and then sells the lime soaps, which contain about 40 per cent., to the proper works. The apparatus required for this precipitation of lime soaps is of an extremely simple nature, and easily obtained. Since lime soaps give rise to the formation of gypsum when treated with

crude hydrochloric acid, which always contains sulphuric acid, and the gypsum makes the separation of the oil from the aqueous liquid very difficult, Vohl experimented with a magnesium salt, by which the fatty acids are as completely precipitated. The magnesia soaps occupy a much smaller volume, contain about 60 per cent. of fat, and produce no gypsum when decomposed by sulphuric acid, or hydrochloric acid containing sulphuric acid. The lime and magnesia soaps may also be directly employed for the manufacture of illuminating gas; the tar, which is simultaneously produced, may be used like pure oil in gas-making. The gas obtained from the lime and magnesia soaps is of very high illuminating power, and the yield is very high.

The residues of oil-manufacturing are recommended by G. F. Reisenbichler to be used in soap-making (*Neue Seifensieder-Zeitung*). After they have been subjected to the strongest pressure, oil seeds retain 5–10 per cent. of oil, which can only be extracted by chemical means. These residues may be used with great advantage in soap-making in the case of very cheap soaps; a small quantity is added, not exceeding 20 per cent. The residues from expressed nuts of all kinds are especially suitable; less suitable, or not at all, are expressed linseed and rape seed, since they contain too many hard particles. When these fatty residues are to be added to soaps, they must be fresh, and previously be finely ground. They are then mixed directly with the lye, which is then boiled, and later the principal fat added to the resulting thick liquor. The saponification is much accelerated by the addition of this powdered material to the lye, and the consequent thick condition of the latter. Reisenbichler recommends in particular for this purpose the residuals obtained in expressing olive oil from the olive. The cake, which contains fairly fine skins and is very oily, readily dissolves in the lye to form a tolerably uniform paste.[1]

Recovery of Glycerine as a By-product.—Glycerine is generally obtained by saponification of fats as a by-product in the preparation of fatty acids. In soap-making small

[1] Elsner regards these additions as not permissible, and soap made in this manner as adulterated.

quantities of glycerine were formerly obtained; according to Schwanert (Muspratt's *Chemie*) the liquid below the supernatant soap—the so-called under-lye—which contains glycerine, alkaline hydroxides, sodium and potassium chlorides, was exactly neutralised with hydrochloric or sulphuric acid, evaporated to dryness on the water-bath, the residue extracted with 90 per cent. alcohol, and the glycerine obtained as a yellow syrup on evaporation of the extract in a still. According to Reynolds, the under-lye was concentrated by evaporation, the salt deposited on the bottom of the pan was removed from time to time and brought into a drainer, from which the liquid could flow back into the pan. When the liquid had reached such a concentration that it boiled at 109° C., it was heated in a still until its boiling-point rose to 193° C., when high-pressure or superheated steam was driven through the upper layers of the liquid and the glycerine distilled over in the steam. It was necessary to regulate the temperature carefully, so that the glycerine should not be decomposed at too high a temperature, and that the distillation should not be stopped by too low a temperature. After the distillation, the distillate was evaporated at a moderate heat, when the glycerine remained behind. When this method of manufacture is considered, it can hardly be supposed that glycerine is now still made in this manner from the under-lyes of the soap-maker, especially since it can be regained more simply, readily, and cheaply from the crude glycerine obtained in the manufacture of stearin candles. But although soap-works may no longer be able to work up the under-lyes for glycerine in this manner, experiments in other directions must not be omitted. Linke first drew attention to the large quantity of glycerine lost in these lyes, which he estimated at 8,500,000 kilos. (8500 tons) per annum in Germany alone, of a value of 3,000,000–3,600,000 marks (£150,000–£180,000). These figures explain the attempts to find a method by which glycerine can be recovered from this liquid, and Schwanert made a step in the right direction when he drew attention to the crystallisability of glycerine, by means of which property it may perhaps be recovered with profit from soap lyes. In 1867, Sarg had

already obtained crystalline glycerine at several degrees below zero, but Kraut, of Hanover, was the first, in 1870, to discover a method of crystallising glycerine in any required quantity and quality. The method was transferred to Sarg's, Son & Co., of Liesing, near Vienna, with all right of priority. This firm obtained patents in Austria, England, and Russia, and worked the process on the large scale. The crystallisation is conducted in sheet-iron vessels, from which the crystals can readily be loosened; they are then freed from the adherent mother liquor by treatment for fifteen minutes in a centrifugal running at 1200 revolutions per minute, and, after they have dried, are melted. Crude glycerine must be once recrystallised. As Schwanert states in these communications, the temperature has a considerable influence on the yield; at temperatures higher than 2° C. the mother liquor amounts to more than 30 per cent. of the glycerine used. In mild winters the crystallisation is not profitable. In addition to the application of a temperature several degrees below zero, a certain amount of movement appears to be one of the conditions under which the glycerine becomes solid and crystalline. Experiments with a view to crystallising glycerine in a simple manner, applicable to larger quantities, would certainly be very profitable, since the most complete purification is obtained in this manner, all the impurities remaining in the mother liquor. The under-lyes of soap-works might by this means be utilised in a most productive manner.

A process for the **recovery of glycerine from waste soap lyes**, due to H. van Ruymbecke, consists in new means for evaporating, distilling, and concentrating these lyes. The apparatus is constituted of cylindrical cast-iron evaporators with the usual accessories—steam-pipes, man-holes, valves, etc. —to which are connected two cylinders, and finally the condenser. The process is as follows:—The soap lye is brought into the evaporator mixed with green vitriol or another suitable metallic salt. Then a low vacuum, about 26 inches, is produced, and steam passed into the cylinder inside the evaporator; a concentration of 28° B. at the outflow, or 30° B. at 15° C., is then obtained. The evaporation causes

the salts to crystallise out, they collect at the bottom, and may be removed, dried, and purified. At the density given above, the liquid contains about 50 per cent. of glycerine; most of the salts have crystallised out. The liquid now passes into a second evaporator, where it is concentrated down to 34° B. at 15° C. Salts again separate, and are removed and dried. The evaporation is effected in two stages, because the salt impregnated with strong crude glycerine would be difficult to dry and free from the mother liquor. From the second evaporator the liquid passes into the distilling apparatus, which works under a vacuum, and in which it is heated almost to boiling by means of steam. Ruymbecke uses again expanded steam, which, injected into the vacuum of about 28 inches, induces rapid distillation. The least volatile distillate collects in the first cylinder, and is very pure glycerine; the greater part of the glycerine is found in the second cylinder; the most volatile distillate, which contains much water, passes into the condenser. If the product is not sufficiently pure, it is distilled again.

The method given by H. Flemming, of Kalk, for the recovery of pure glycerine from soap lyes, depends on the application of dialysis (German patent, 12,209). Glycerine is a substance which, when in aqueous solution, cannot pass through parchment paper, whilst the salts contained in the under-lyes can readily penetrate. In order to separate the glycerine from the salts, Flemming employs an apparatus very similar in construction to the diffuser used in sugar-works. The lyes, previously evaporated until much salt separates, flow through vessels, one side of which is made of parchment paper; water flows on the other side of the parchment paper. The process which occurs is an ordinary osmosis; the salts in the glycerine solution pass through the permeable division —the parchment paper—and dissolve in the water, a corresponding quantity of which passes through to the glycerine and dilutes it.

In working with under-lyes, which are strongly alkaline, it is advisable to neutralise with sulphuric acid before dialysing, since the sulphate diffuses more rapidly than the compounds of alkaline reaction. When the under-lye is sub-

jected to the osmotic action for a sufficient length of time, the glycerine solution is obtained very pure, when it can be converted into pure glycerine by concentration, at first in open pans, then by distillation in a vacuum apparatus. The low cost of providing an apparatus suitable to the dialysis of under-lyes, which would consist essentially of a vessel containing frames stretched over with parchment paper, makes Flemming's process available even for small works, which can then utilise very well the under-lyes which would otherwise be wasted, and dispose of the dialysed solutions to glycerine works for further treatment.

CHAPTER XLVIII

ALKALI WASTE AND THE RECOVERY OF SODA

Utilisation of Alkali Waste.—Since almost all soda-works make sulphur from alkali waste, there remains a residue consisting chiefly of carbonate, sulphate and sulphite of lime. According to M. Schaffner (*Dingler's Journ.* 199, p. 243) this residue, from which railway embankments can be made, is especially suitable for the upper layers, where it is used instead of, or together with, river sand. The sleepers lie dry, since the desulphurised residues are porous; the latter also appear to have a considerable preservative action on the sleepers.

Schott proposed to use alkali waste in glass-making, since the sodium sulphide would serve as a reducing agent in place of coal, and lime would also be introduced into the mixture. O this point, however, Lunge remarks (*Dingler's Journ.*, 216, 375): "Alkali waste always contains, in addition to calcium sulphide, excess of lime both as carbonate and caustic lime, and excess of coal, and would thus be the more suitable for the use proposed by Schott if it did not contain, with these useful constituents, certain harmful substances — alumina, ferric oxide, gypsum, etc." These constituents prevent the use of alkali waste except for the lowest qualities of glass; and, in fact, Lunge himself disposed of alkali waste to makers of beer bottles. The desulphurised residue, treated by the process of Schaffner or Mond, which is never free from undecomposed sodium sulphide, is suitable for the same use.

Recovery of Soda in Cellulose Works.—In regard to the recovery of soda in cellulose works, M. Faudel remarks in a long paper (*Dingler's Journ.* 219, p. 432), in the first

place, that, generally speaking, in the best works the recovered soda amounts to little more than 70 per cent. of the quantity used. Although the lime mud obtained in causticising the soda be repeatedly washed with fresh water, and the dilute liquors obtained be used in preparing strong liquors, and although the most rational apparatus be used for washing the boiled wood, yet there is lost in the lime mud about 5 per cent., in the wood the same quantity, by leakages and in the evaporating furnaces about 20 per cent.; thus, on the whole, about 30 per cent., and in some works, no doubt, much more.

Faudel assigns a portion of the blame for this condition of things to the evaporating furnaces, which, in some works, are long, low flues with a powerful and wasteful fireplace at one end, from which the fire gases pass over the lyes. The liquors are evaporated in these furnaces only to a certain concentration; they are then removed in the form of a tolerably solid but tarry mass, and further heated and melted on separate hearths or on iron plates in the open. The soda is thus obtained in the form of large hard lumps, which frequently have to be broken and ground, at no inconsiderable cost, before they can again be used in the preparation of the lye. Quite apart from the complexity of the process, enormous quantities of heat are lost, owing to the formation of a solid crust over the surface of the lye in the furnace, which there is not always a labourer to break up at the proper time, and which hinders the further evaporation of the liquor beneath.

According to Faudel, Porion's evaporating furnace is more rational than the one just described; the soda it produces is porous, and may be used in the preparation of fresh caustic lye without pulverising. A furnace used in South Germany permits the liquor to flow down a series of pans arranged in steps, so that it meets the fire gases on its way. This furnace, which is said to require frequent repairs, produces the soda in as inconvenient a condition as that first described, and probably, therefore, offers little advantage over it.

A fourth furnace, which appears to the author as worthy

of notice, is that patented by the Belgian Werotte, and known under the name of Fernau.

M. Montagnon, a Paris chemist, has proposed a method for the recovery of soda in the wet way (*Journ. des Fabricants de Papier*), which simply consists in removing from the black liquors (of cellulose works) the gummy, albuminous, and mineral matters derived from the plants. This can only be done when they are precipitated as compounds insoluble in a combination of the liberated soda with the precipitant. The compound of soda and the precipitant remaining in solution must then be treated with a base which expels the acids combined with the soda and gives the soda alone, dissolved in the water used at the first solution. These conditions are fulfilled by certain common and cheap materials—pyrites and lime, or powdered iron ore (ferric oxide) and sulphuric acid. The cost of the recovery of 1 ton of soda is given below for two methods of procedure, in which either (1) pyrites and lime, or (2) ferric oxide and sulphuric acid are used.

The cost of the recovery of soda in the wet way is not to be regarded as pure loss, since certain by-products in great demand are also produced. These by-products may be used with advantage for bleaching paper-pulp, or may be converted into the original materials and so used over again.

In using pyrites and lime to recover the 1 ton of soda contained in 15,400 gals. of black liquor, 2 tons of calcium sulphite are formed, from which the sulphurous acid may be obtained by treatment with sulphuric acid and used for bleaching paper-pulp.

When ferric oxide and sulphuric acid are used to recover 1 ton of soda, 2½ tons of ferric sulphate are produced, which can either be sold as such or decomposed in reverberatory furnaces into ferric oxide and sulphuric acid, which may then again be used in the recovery of soda.

In both cases there are produced about six tons of the extracted gummy, albuminous, and mineral matters, the mixture of which is sold as a fertiliser at a value of ten shillings per ton.

The following calculations of the costs make clear the relative advantages of the two processes of recovery:—

1. Recovery in the Dry Way (Porion's Process).

	s.	d.
3·7 tons of coal at 22s. 6d., for the evaporation of 70 tons of water	82	3
1 ton of lime for causticising	20	0
Loss of soda, 20 per cent. from 1 ton, *i.e.* 4 cwt., at 28s.	112	0
Depreciation and interest for the furnace, *i.e.* 15 per cent. on £1600, for a daily recovery of 1 ton for 360 days per annum	13	4
Total	227	7

2. Recovery in the Wet Way (Montagnon's First Process).

			s.	d.
1·25 ton of pyrites, at 24s.			30	0
1 ton of lime			20	0
Wages and general expenses			40	0
4 cwt. coal at 24s.			4	10
Loss of soda, at a high estimate, 20 per cent. from 1000 kilos., *i.e.* 4 cwt., at 28s.			112	0
Depreciation and interest, 15 per cent. of the cost of the installation, £200, *i.e.* per day			1	8
Total			218	6
Value of by-products to be deducted—	s.	d.		
6 tons fertiliser at 10s.	60	0		
2 tons calcium sulphite at 32s.	64	0		
			124	0
Cost of recovery per ton			94	6

(Montagnon's Second Process.)

			s.	d.
1·13 ton of ferric oxide at 20s. per ton			22	7
1·58 ton of sulphuric acid at 4s. per cwt.			126	5
Wages and general expenses			40	0
Loss of soda, at a high estimate			112	0
Depreciation and interest			1	8
Total			302	8
Value of by-products to be deducted—	s.	d.		
6 tons of fertiliser at 10s.	60	0		
2·5 tons of ferric sulphate at 56s.	140	0		
			200	0
Cost of recovery per ton			102	8

The economy resulting from Montagnon's processes over the recovery in the dry way is therefore, for 1 ton of recovered soda :—

1st process . .	227s. 7d. – 94s. 6d.	= 133s. 1d.
2nd process . .	227s. 7d. – 102s. 8d.	= 124s. 11d.

Thus for every cwt. of straw treated there is a saving of eight shillings, or two shillings on one cwt. of white paper-pulp.

Recently (*Civilingenieur*), R. Schneider has constructed an evaporating and calcining furnace for the recovery of soda in cellulose works, which appears worthy of special mention. The waste liquors of cellulose works are evaporated and the residue calcined without the production of noxious odours, by the application of highly heated air. In 1875 Siemens described a regenerative hot-air apparatus, consisting of two chambers, built of fireproof bricks, with the requisite openings for air to enter and leave, and filled with fireproof bricks after the manner of the well-known Siemens' regenerators. In Schneider's apparatus two of these chambers are connected with the calcining and evaporating furnaces. The larger stands in front of the furnace, which is somewhat smaller, about 17 metres (56 ft.) long, at a distance of about one-third of the length of the furnace from the principal apparatus. The gas obtained from lignite in the producer —a simple vertical furnace—passes through flues provided with dampers into the hot-air apparatus, where it burns, the resulting products of combustion being then conducted, not to the calcining furnace, but by flues direct to the chimney. By the proper change in the dampers a current of highly heated air from the larger apparatus enters the furnace at the end, and a second current from the second hot-air apparatus enters, in a direction at right angles to the first, the calcining space just in front of the fire-bridge. The gases pass under the wrought-iron evaporating pan, then between this and a second evaporating pan, and finally under a preliminary heater, from which they pass to the chimney, 35 metres (115 ft.) high. The liquor collected in the preliminary heater passes in the opposite direction through a pipe into

one evaporating pan, thence to the second, and finally to the calcining furnace. The calcining space has a capacity of about 2·7 cub. metres (95 cub. ft.) of liquor, one evaporating pan a capacity of 7·5 cub. metres (265 cub. ft.), the other 17 cub. metres (600 cub. ft.), and the preliminary heater 29 cub. metres (1023 cub. ft.).

If we assume that the hot-air apparatus is in the prescribed condition and is supplying a continuous current of highly heated air to the calcining space, then the process is conducted as follows:—The evaporating pans and preliminary heater being full, the valves are opened, the calcining space slowly filled, and the two evaporating pans again filled according to the indications of the gauge glasses. According to the consistency of the liquor, this process is repeated two to three times within three to five hours, during which time heating gases enter from the larger apparatus, in consequence of which the temperature in the calcining space rises considerably, despite the strong evaporation. When the evaporation has proceeded to a certain extent, after about three hours the current of gas is cut off, and there now enters into the larger apparatus, as during the whole period already from the smaller hot-air apparatus, only atmospheric air at a high temperature (about 1000° C.). This air is thus present in the required quantity from the commencement of the calcination and is completely diffused through the whole of the calcining space. It would therefore appear to be impossible for any gas of organic origin to leave the calcining space undecomposed.

When the mass in the calcining furnace is completely burnt, it is drawn out through doors, and, after removing the iron plates, brought into the cooling-room. The gases which are still produced here during the slow cooling enter the calcining space through openings provided for the purpose, and burn there completely in the hot current of air coming from the larger hot-air apparatus.

CHAPTER XLIX

WASTE PRODUCED IN GRINDING MIRRORS

Utilisation of the Polishing Sand.—In grinding glass, especially in making mirror-glass, a very fine polishing sand is used, which, after it left the apparatus, was formerly useless. The pasty consistency, and absence of plastic nature, were great obstacles to further treatment. Le Motte, a Paris engineer, has obtained a patent for the use of this waste in making a very hard building-stone. The mud from the grinding-tables, consisting of fine glass dust, quartz flour and finely divided cast-iron, is first run into settling-tanks, from which the supernatant water can be removed as required. The deposit is then dried in centrifugals, mixed with fine clay powder or glass flour in a mixing and kneading-machine, pressed into moulds, and burnt at a high temperature to a very hard mass. A pressure of 300 kilos. per sq. cm. (4220 lb. per sq. in.) is required, since the bricks are very porous. These bricks are not attacked by most mineral acids, they also exhibit a very considerable resistance to chloride of lime and sodium sulphate. The weight is not more than that of ordinary porous bricks. By mixing the sand, enamelled bricks may also be made, suitable for the decorative treatment of façades.

Another account states: The used grinding sand, containing more or less clay, which is produced in considerable quantities in grinding mirrors, is to a great extent in such a fine state of division that it has become plastic and can be moulded whilst wet. The fine particles of glass, which have been ground off, make the mixture still easier to mould; the sand may thus not only be moulded into various shapes, but the objects produced can be burnt. The quantity of the fusible sodium silicate in the used sand is so considerable that

it is sufficient, with proper burning, to cement together the grains of quartz, and thus impart to the moulded object a certain degree of strength.

The moulding, pressing, drying, and burning are done in the ordinary manner; hard bricks, hollow or solid, plain or ornamental tiles, building-stones and architectural ornaments are readily obtained. The product, which is very light—the specific gravity is about three-fifths of that of ordinary stone—exhibits no small resistance to mechanical and chemical actions.

CHAPTER L

WASTE PRODUCTS OF THE MANUFACTURE OF STARCH

Utilisation of the "Fruit Water."—In working by the ordinary process by which starch is obtained, the potato pulp coming from the grinding-machine is diluted with so much water that the proportion of valuable constituents appears to be reduced to such an extent that they cannot be utilised (F. Rehwald, *Die Stärke-Fabrikation*, Vienna, Hartleben). This "fruit water," if it has not fermented, may be suitably employed as a direct addition to pigs' food. If steam be available, it may be passed into the "fruit water" until the albuminous matters have coagulated into flocks, which are separated by running off the liquid into the drain, and used as cattle food.

Utilisation of the Pulp.—This mass is most suitably mixed with other cattle foods; since it is somewhat difficult of digestion, it is advisable to break it up by heating to boiling. The pulp, however, still contains so much starch that it is profitable to recover it. By suitable treatment 2–2·5, or even 3 per cent., of the total quantity of starch may be recovered from the pulp, thus producing a considerable increase in the profits. The pulp still contains many uninjured starch cells which have escaped the grinding. In order to rupture these cells, a simple arrangement is employed which drags the cells lengthways and thus ruptures them. The apparatus is constructed as follows:—A roller of polished marble or granite is connected with a cog-wheel having a certain number of teeth. Below this roller, but very near to it, lies a second, also connected to a cog-wheel, which has, however, only about one-third of the number of teeth on the upper roller. Thus the upper roller makes three revolutions

whilst the lower is making one. The pulp in passing between the roller is at the same time squeezed and drawn out in consequence of this unequal velocity; thus almost all the starch cells contained in it are ruptured. When the rolled mass is brought into the cylindrical brush apparatus starch can be obtained from it.

The process is generally conducted by collecting a quantity of the pulp, then rolling it and treating it alone. The starch which has been semi-dried and purified by settling also produces in this case a certain quantity of starch mud, which is treated either separately or together with similar material. In places where there is a distillery it is advisable to work up the pulp with the other material for spirit; thus all the starch is extracted, and the cork substance goes into the distillery wash.

If the pulp is collected into heaps, which are kept moist, a process of decomposition soon sets in and gradually but completely destroys the cork. The whole mass changes to a soft cheesy mass, consisting of the decomposed cork substance, starch, and cellulose. If it be then brought into the apparatus for obtaining starch, the latter is obtained pure. For this purpose the pulp is allowed to lie in thin layers for such a time that it is still moist at the top, when heaps are formed, 60–80 cm. high (24-32 in.), resting on a layer of boards and covered by twigs or boards. After a few days the temperature of the whole heap rises, the covering of boards protects it from cooling, and the fermentation begins. In order to give the air as much access as possible, it is advisable, in building the heaps, to insert poles and to draw these carefully out, so that channels may be formed into the interior of the mass. Repeated turning over with shovels and, if necessary, moistening with water, greatly accelerate the fermentation of the mass.

If the formation of gases of bad odour is remarked during this process, it is a sign that another process of fermentation is taking place in addition to the decomposition, by which the yield of starch would be diminished. A buttery or cheesy smell denotes the occurrence of the butyric fermentation, by which the starch itself is attacked. This incorrect course of

the process is due to the entrance into the mass of too little air; the mass is either too compact or too wet. The evil may be combated by shovelling over the mass, or by allowing it to dry.

Utilisation of the Gluten.—In the fermentation process the gluten is destroyed with the exception of a small quantity, which rarely exceeds 25 per cent. In the process without fermentation, the yield of gluten may amount to more than 90 per cent. of the total quantity. In addition to the use of gluten as cattle fodder, for which purpose the gluten obtained by the process without fermentation is stirred with water and heated to 70° C., it is also used in making foods. In the latter case, the first operation is to obtain gluten-flour, as described by F. Rehwald (*loc. cit.*).

Wheat, from which the husk has been removed, is thoroughly washed; the starch layers obtained are not subjected to a further washing, but are partially dried by the air-pump and mixed with gluten. The mass is then made plastic by an admixture of potato-flour, instead of which dark-coloured wheat-flour or pea or bean-flour may be used. It is of great importance to mix the gluten most intimately with the starchy substances, for which purpose a kneading-machine is employed, as used by bakers. The mixing may also be done by means of rollers, in which case a first pair of rollers produces a band of a certain thickness, which is stretched out more and more by the succeeding pairs of rollers. The thinnest band is collected, pressed together, and again subjected to the action of the rollers; there is thus obtained by repetitions of the process a more and more uniform mixture of gluten and starch. It is advisable to add to the mass, before kneading, $\frac{1}{1000}$ of its weight of sodium phosphate and $\frac{2}{1000}$ to $\frac{4}{1000}$ of common salt. The paste is filled into a shallow rectangular box, with a narrow slit at one end, through which it may be expelled by a piston. Below the box is a long endless belt of linen cloth, running over rollers. When the paste is expelled through the slit in the box by means of the piston, it leaves in the form of a thin band, which is carried away by the endless cloth, from which it is taken on to boards. It is then dried in the sun to a thin, horny mass, which can be easily broken and ground to flour. This gluten-flour far

surpasses beans in nutritive value; a food is obtained from it by stirring into boiling water, to which salt has been added.

The following process is used in order to obtain gluten in the form of groats. The gluten, made into a paste with potato flour in the above-mentioned manner, is brought into a cylinder fitted by a piston, and closed at the bottom by a metal plate perforated by the largest possible number of holes. The holes are slightly conical, being about 1·5 mm. in diameter at the top and 1 mm. at the bottom. By applying pressure to the piston, the paste is made to issue in the form of thin threads, which are then cut up into small pieces by a knife below. The apparatus is arranged so that pressure is only applied intermittently, in order that the grains may be as far as possible of equal size. Below the cylinder there moves slowly a stretched endless cloth, which catches the grains and carries them away; they are then lightly dusted with flour. In order to round the grains, they are brought, together with 5 per cent. of potato-flour, into a barrel, through the bottom of which passes an axle, and which is closed by a door in the place of the bung-hole. The grains are rounded by the slow rotation of the barrel; finally they are sorted through sieves of different meshes, and dried in thin layers on boards.

Utilisation of the Residues.—G. Thenius recommends a new method for the utilisation of the residues obtained in the manufacture of starch (*Chem. Zeit.* 150; Post's *Zeits. f. d. chem. Grossgew.*, III. 2, 280), which consists in evaporating the wash waters—the "softening" waters—which have previously been run to waste. By evaporating 10 grms. of this liquid, Thenius obtained 2·09 grms. of extract, which was of a pale brown colour, had a sweetish, salty taste, and an odour similar to that of malt extract. If the extract is dissolved in a little water, the less soluble salts—sulphates, phosphates, sodium, potassium, and magnesium chlorides—separate. The portion soluble in water is again evaporated to dryness on the water-bath; it then possesses a pleasant, sweetish taste, but is not salty. One hundred parts of the extract contained 25·6 parts of salts and 7·44 parts of pure extract. From a vessel of 15 hectolitres (330 gals.) capacity, 30 kilos. (67·5 lb.) of extract are obtained. The extract can be profitably employed

in bread-baking or in making cattle foods. The bread prepared with it is said to have an agreeable flavour, and to keep moist for a long time. The purified extract can be used medicinally, and as a substitute for malt extract. The extract is obtained by evaporating the liquor in shallow pans by means of the waste steam from the boiler.

Utilisation of the Waste Waters.—In most cases the waste waters are run to waste; they then frequently give rise to expostulations and complaints, since the decomposable matter they contain ferments, and may thus taint the neighbourhood and injure the fish in the rivers.

As long ago as 1835, Burggraf made successful experiments on the use of this water for irrigating meadows and arable land (*Polyt. Journ.*, 56, p. 464), and in 1877 Märcker gave an account of comprehensive experiments on the irrigation of meadows by its means (*Zeits. d. landw. Centralver. d. Provinz Sachsen; Dingler's Journ.*, 225, p. 395). The waste water, which is first run through two settling-tanks, in order that the fine grains of starch still suspended in it may be deposited, is conducted for a distance of 170 metres (185 yards) through a pipe 15 cm. (6 in.) wide, with a fall of 33 mm. per metre (3·3 in 100). The water is received from the pipe in an open ditch 1 metre (39 in.) wide and 30 metres (33 yards) long, which conveys it to a small storage pond. It is here mixed with pure well-water, in order to avoid the strong concentration of the original waste water, and to obtain larger volumes of water for regular distribution over the meadows. The mixed waters are then carried to a large meadow of 7½ hectares (18½ acres), over which it is distributed by a system of large and small ditches, and retained by dams. The water is conveyed away by a system of drains, which can be put in or out of action by sluices as required. The drainage then goes to a second meadow of 5 acres, and thence to a third of 6 acres. The crops obtained are described by Märcker as excellent.

Märcker observes, in conclusion, that the waste waters of starch-works may be applied to the irrigation of meadows with the greatest advantage, because there is not only an increase in the crop, but the composition of the hay is also considerably improved.

CHAPTER LI

STEARIC ACID

Recovery of Waste Stearic Acid.—The process described is due to Weiss & Co., and is patented in France. The oleïc acid after it leaves the press is subjected for a longer or shorter period to a well-arranged system of cooling, so that its temperature is maintained constant at about 5° C. At this temperature almost the whole of the stearic acid separates from the oleïc acid. The mass, which has thickened, is transferred as quickly as possible to a centrifugal machine, the drum of which is protected by wool. The centrifugal is rotated at 1200–1300 revolutions per minute, when the oleïc acid is separated within a few minutes from the stearic acid crystals. The residual stearic acid in the drum is then pressed in the ordinary manner. For this purpose the centrifugal machines used in sugar-works are quite suitable. The cooling of the oleïc acid is effected by cooling a relatively considerable quantity of water to 4–5° C. by means of an ice-machine, and then circulating it through a system of pipes, which surround or traverse the tank containing the oleïc acid.

CHAPTER LII

VEGETABLE IVORY WASTE

Utilisation of the Waste of Ivory-Nuts.—Guild, of New York, makes moulded objects of all kinds, especially buttons, from the waste of ivory-nuts. The process is as follows:—The waste is ground to a fine powder and the mineral particles removed by stirring up the powder with much water, when the mineral constituents rapidly sink to the bottom, whilst particles derived from the nuts remain suspended. The water is then drawn off together with the suspended powder, which is separated by filtering and pressing, and then dried in any suitable manner. The dry powder is sieved and mixed with enough water to make the separate particles cohere. The process may also be simplified by drying the powder only to such an extent that it retains the proper quantity of water. The mass obtained is filled into moulds, which give the shape of the desired article, and pressed in a press whilst subjected to a temperature of 125–150° C. According to the size, the moulds are subjected to the action of pressure and heat for a period of three to ten minutes. The pressed articles are then taken out of the moulds, dried, and finished. In making coloured articles, a colouring matter is added to the mass before it is filled into the moulds.

Brolik employs ivory-nut waste as a hardening material for Bessemer steel and iron. The hardening powder is made from the sawdust and the smaller turnings and filings. The larger pieces can be ground in a drug mill or other similar apparatus, yet it is advisable to employ instead a suitable rasping arrangement, since there is then a considerable saving in cost—ten to twelve shillings per cwt. being charged for grinding the shavings. The powder obtained in this manner may also be mixed with other hardening agents. The latter substances can also, if they are soluble in water or

at least able to impart their special properties to it, be made to penetrate the powdered portions of the nut, by placing the powdered nut, which strongly attracts water, in water containing the requisite substances, removing it after twenty-four hours, and thoroughly drying it. Hardening experiments were conducted in the following manner with unmixed ivory-nut powder, ground to the size of fine sand:—A cementation box, filled in the ordinary manner with Bessemer steel plates and iron to be hardened, and with leather charcoal, received at the top at one narrow end a layer of the ivory-nut powder, in which were placed a disc about 5 cm. (2 in.) in diameter and then a piece of steel about 12 mm. (½ in.) thick. The lid was then placed on the box and the joints luted with clay. After heating in a coal fire for three hours the box was opened. When cold it was found that, in consequence of insufficient or irregular heating, insufficient time or bad leather charcoal, the iron placed in the latter was irregularly hard, so that it could not be used and had to be hardened again, whilst the two pieces in the ivory-nut powder were of uniform hardness and could not be attacked in any place by a good new file without blunting it. The disc was then broken; it showed on all sides a completely regular hardened crust about 1 mm. (·04 inch) thick. The hardening experiment was then repeated with fresh ivory-nut powder; in regard to the hardness, exactly the same results were obtained. Although the hardness of the iron immersed in the ivory-nut was faultless and of a regularity not hitherto obtained, it was found that ivory-nut powder of too fine grain was not suitable for hardening steel which was required to be of good appearance. The surface of the hardened steel was covered with spots, which were presumably produced by the penetration of the exceptionally fine grains of powder into the pores of the iron when enlarged by heat, where they remained and were fixed by the contraction during or after cooling. It is not impossible that with coarser powder, as from the sawdust and raspings, or by mixing or saturating the ivory-nut powder with other suitable ingredients, this hindrance to the general use may be avoided.

CHAPTER LIII

TURF

Utilisation of Turf Waste.—G. Gercke, jun., of Hamburg, has patented a process for producing plastic objects from turf as a substitute for wood, pasteboard, paper, etc. This process is based upon obtaining the turf without damage to the fibres, which are easily injured whilst moist. In removing the turf, in order not to cut through the fibres, the longitudinal layers must be followed, which occasionally form flat sheets.

After the raw material has been cut to the desired dimensions, it is dried upon boards or a framework of laths, during which it must be protected from the sun in order to prevent the plates from warping. When completely dry, the material is subjected to a special treatment, the essential part of which is strong compression, according to the particular purpose for which it is required. The very hygroscopic nature of the product must be removed if it is to be exposed to the action of the weather. For example, in order to make a roofing material, the dry turf plates, compressed to 10–20 per cent. of their original thickness, are protected from moisture by saturation with a warm mixture of tar and asphalt. The excess of tar is removed by squeezing between rollers and the plates then dusted with sand in order to prevent them from sticking together.

Another method consists in saturating the turf plates with milk of lime, drying, pressing or rolling, soaking in water-glass, and then coating with water-glass paint.

In order to increase the strength of these plates, which is already considerable, materials of various kinds are inserted and fastened between two or more turf plates by means of a suitable adhesive agent. For this purpose are used:

bast, tow, cord, long turf fibres, heather, twigs, metal, waste wood. The binding and impregnating materials are: tar, asphalt, wood cement, glue, water-glass, varnish, pitch, clay, rosin, cement.

The compressed turf may be subjected to any required treatment; it is suitable for replacing papier-maché and wood-pulp, wood, horn, bones, etc. (Compare T. Koller, *Die Torf-Industrie*, Vienna, Hartleben, pp. 109 *et seq.*)

CHAPTER LIV

THE WASTE WATERS OF CLOTH FACTORIES

Utilisation of the Waste Waters in Cloth Factories.—It is well known that the waste waters foul the streams into which they run, and are, on that account, often a source of great inconvenience, and frequently a restraint on the manufacture. This is especially the case in flat countries, such as Holland, where—as, for example, at Tilburg—the endeavours to remove the stagnant waste waters, in this case on sanitary grounds, have been extraordinary. According to the method of E. Schwamborn, of Aix (*Dingler's Journ.*, 216, p. 517), the waste waters of cloth factories are used to prepare lime soaps. The process follows.

Attempts at clarification, by means of sand or cinder filters, in the so-called clarifying vats, have been unsuccessful, on account of the mechanical difficulties in separating the soap, though they may have succeeded with the solid suspended impurities. The soap may be removed by chemical means, when not only is the water made clear, but also the fatty matters contained in it are regained. Schwamborn's process is applied in a similar manner to the recovery of the wool-fat from the waste waters of wool-washing. The waters used in fulling and rinsing the cloth are included under the head of waste waters. They contain the oil used in spinning, to the extent of 15 per cent. of the weight of the yarn; the soap used in fulling, up to 30 per cent. of the weight of the cloth; also the size used for strengthening the warp, and, in addition, dissolved colouring matters and wool fibres. The clarification of these wash waters depends on their decomposition by means of milk of lime; the method is here described:—

The washing-machines are provided with two waste pipes—the one to conduct the first thick liquid, which is gradually

becoming more dilute, into the collecting-tank; the other, for the direct removal of the next clear waters, which are fit to run direct into a stream. When the collecting-tank, the capacity of which is assumed at 150 cub. metres (5295 cub. ft.), is full, which takes about fourteen days with a consumption of 2000 lb. of soap, corresponding to the washing of about 8000 lb. of cloth, its contents are run off through a pipe at the bottom, into a tank of equal size at a lower level. At the same time a thin stream of milk of lime is run from a vessel (a vat provided with a tap) at a higher level, into the outflow pipe, so that intimate mixing is obtained. Sloping ground is an advantage for this arrangement; when the slope is absent its place must be taken by pumps.

The bottom of the second tank—the decomposing tank—is made of three layers of bricks. The lowest is continuous; in the next, the bricks are laid with as great interspaces as the third layer, which is also continuous and united with mortar, will permit. This system of drains has an inclination towards one corner of the tank, and communicates there with a wooden funnel, firmly fixed over a drain, reaching to the top of the tank, and provided with a slanting series of holes, which are closed with wooden plugs whilst the suds are being admitted.

The decomposition takes place immediately the liquid enters the tank; the lime soap separates in flocculent form, surrounds the solid suspended substances, such as colouring matters, wool fibres, etc., and sinks with them gradually to the bottom, where it finally unites to a thick precipitate. Even in a few minutes the uppermost layer of the liquid is free from the precipitate, and not only clear, but also colourless. This clarification, which extends to the dissolved as well as the suspended colouring matters, is so energetic, that considerable quantities of dye waters may be added and simultaneously purified. The characteristic appearance of the flocks in the clear water affords a sign of a sufficient introduction of lime; excess, however, does not hinder the purification. For 150 cub. metres (5295 cub. ft.) of the suds, there is required approximately 0·3 cub. metre (10·6 cub. ft.) of lime paste, in the condition in which it

occurs in the lime pits; this quantity naturally varies according to the amount of soap in the suds.

The clear water is run off by drawing the wooden plugs, fixed in the funnel, from above down to the point to which the thick lime soap is found to have settled. The manipulation is easier if a board partition, also provided with plugs, be placed transversely in front of the funnel, up to half the height of the tank. More water is removed, partly by evaporation, which is assisted by the cracking and shrinking of the mud, and partly by filtration in the system of drains at the bottom. After several days, the substance lying at the bottom is a dried fissured paste, which is thrown out on the edge of the tank, and spread, in order that it may dry further. In winter, when necessary, the final drying takes place under cover on suitable supports. If the space at disposal permits a second decomposing tank to be laid down, the drying is made considerably easier, by reason of the double time allowed.

The lime soap retains the last portions of liquid for a long time, whilst, on account of its fatty nature, *i.e.* its want of adhesion to water, it does not again take up water, such as rain, which may reach it. A lump, after drying in the air, may lie for days under water without appreciably gaining in weight. The residue from a tank 1·5 metre (4 ft. 11 in.) deep, is about 6 cms. (2·4 in.) thick, which corresponds to 4 per cent. of the liquid.

From statistics it is calculated that the yearly quantity of cloth washed in Europe is about 10,000,000 cwt. Now, 8000 lb. correspond, as stated above, to 5295 cub. feet of water, or to 2000 lb. of soap, and include 800 lb. of oil used in the spinning (calculated at 10 per cent. of the weight of the cloth). There are obtained on the average about 1600 lb. of lime soap. Accordingly the soap waters of Europe correspond to about 2,000,000 cwt. of lime soap per annum, which is produced from 2,500,000 cwt. of soap, containing 45 per cent., or 1,125,000 cwt., of fatty acids, and from the oil used in spinning, 10 per cent. of the weight of the wool, or 1,000,000 cwt. of fatty acids. Thus, there is a total of 2,125,000 cwt. of fats annually used in European cloth factories.

The lime soap is insoluble in water; it dries to a pale to dark-grey substance, which can easily be cut, has a fatty feel, and burns with a flame when lighted. By decomposing the lime soap with acid, and then washing with hot water, a fatty substance is obtained, which may be directly subjected to distillation. The distillate separates on pressing into solid and liquid fatty matter. According to Stahlschmidt, if the lime soaps are decomposed, and then treated with ether or carbon bisulphide, fatty acids are obtained which are at once applicable in soap-making. Treatment of the lime soap in this inexpensive manner would be very profitable.

CHAPTER LV

WINE RESIDUES

Utilisation of the Marc.—This utilisation extends to the production of tartaric acid, the preparation of marc brandy and œnanthylic ethers, the production of illuminating gas, Frankfort black, and grape-seed oil. To these are to be added the methods used by wine-growers for utilising the marc: the production of marc brandy and after-wine, the preparation of wine vinegar from the marc, the use of the marc in making verdigris, the consumption of the marc by cattle, its use as fuel, and the recovery of potash from the ash.

Utilisation of the Crude Tartar.—To this division belong the preparation of tartaric acid from crude tartar and calcium tartrate, the preparation of cream of tartar and the more important tartaric acid compounds.

Utilisation of the Lees.—The principal products are: lees brandy and œnanthylic ether, calcium tartrate and cream of tartar, Frankfort black, and lees wine.

We must here abstain from dealing further with this highly important industry of waste products, which is so extremely profitable in wine-growing districts, since a special work on the subject has just been published, which is highly to be recommended. In it the utilisation of all the wine residues is taught in so thorough a manner, easy to understand and highly practical, that the whole work would have to be reproduced if we were to deal with the subject in a thorough and useful manner. We therefore refer anyone who intends to undertake the treatment of wine residues, which is extremely profitable in wine-producing districts, for instruction in the necessary arrangements and method to the book *Verwerthung der Weinrückstände*, etc., by Antonio

dal Piaz, Vienna, Hartleben. To this practical work we add here, in regard to the treatment of wine lees, only the process by which the firm of Wagemann, Seybel, & Co. of Liesing, near Vienna, produces tartar and tartaric acid from this substance.

According to E. Kopp (*Naturf. Ges. in Zurich; Neueste Erfindungen und Erfahrungen*), 4000 cwt. of tartaric acid are produced by this firm. The wine lees, which deposit separately in the spring after the main fermentation, amount to about 5 per cent. of the wine. They were generally only used as manure. Since 1854 Seybel has successfully endeavoured to facilitate the collection. The process begins by subjecting the lees to a high pressure in filter presses, 100 eimer (58 hectolitres or 1276 gals.) then produce 40 hectolitres (880 gals.) of wine and 20 cwt. of dry lees. With the yearly production of about 4,000,000 eimer of wine in Austria, the lees deposited amount to 60,000 cwt., and represent a value of over 4,000,000 francs (£160,000). The pressed lees contain organic matter, yeast cells, etc., tartar, tartrate of lime, colouring matter, clay, and sand; they are treated with hot dilute hydrochloric acid, which dissolves principally the tartar and tartrate of lime. The liquid, after filtering through woollen cloth, gives, on cooling, crystals of tartar, which can be purified by recrystallisation. The mother liquors, when neutralised by milk of lime, give a precipitate of tartrate of lime, which is converted into tartaric acid. From the last impure mother liquors of the tartaric acid, a further quantity of tartar is obtained by the addition of potassium chloride.

A process introduced by Müller and Schlosser, of Vienna, for the recovery of tartaric acid from the deposits in discharging vats, is allied to the above treatment of wine lees. It is well known that tartaric acid is used in considerable quantity for discharging printed Turkey-red fabrics; it is then precipitated in a bleaching-powder vat as tartrate of lime. The method of treatment is not known, but is readily imagined. It would be very convenient to combine it with the treatment of wine lees. The calcium tartrate from the discharging vats contains excess of lime, which could

serve for neutralising the hydrochloric acid decoction of the lees.

Among the applications of grape marc Pasque gives attention to the production of oil (*Giorn. agr. ital.*), which, according to Pezeyre, is very simple and profitable. The well-dried marc is stirred to a paste, brought into a pan, frequently well stirred, boiling water added—1 gal. to 4 gals. of marc—and, as soon as the oil exudes, the whole mass brought to the press. The oil is pale yellow and odourless; it can be used for burning purposes, and, when sufficiently purified, for culinary use, but it should not be long kept, since it soon becomes brown and rancid. The oil cakes may find application as fuel in distilleries; the ash forms an excellent manure for vineyards, being very rich in potash.

The firm of D. Savalle fils et Cie., of Paris, has constructed a special portable apparatus for the production of alcohol from grape marc (*Oesterreich-ungarische Brennerei-Zeitung*). Although this apparatus cannot be taken to every vineyard on an ordinary waggon, yet it can be taken to some one vineyard, to which the marc from the neighbouring vineyards can be inexpensively and simply removed. Not only is the pressed grape marc to be distilled, but also the fluid residues obtained after the fermentation and repose of the wine, the deposit, and the lees. With such a method, the co-operative spirit is a necessity, for several, or many, vine-growers would have to combine to obtain the apparatus; they would then treat their residues in turn according to agreement.

CHAPTER LVI

TINPLATE WASTE

Utilisation of Waste Tinplate.—A. Ott, of New York, has obtained an English patent for utilising waste tinplate (*Deutsche Industrie-Zeit.*). The tin is recovered from the tinplate by means of a mixture of acids, which also dissolve the lead contained in the coating on lower qualities of tinplate. From this solution the lead is precipitated, and then the tin by means of sheet zinc; the tin is washed with water and melted. The acids also dissolve about 5 per cent. of iron; for every two parts of tin about one part of zinc is dissolved; the liquid obtained, therefore, consists of a mixture of iron and zinc salts. It can be used for saturating wood, as a disinfectant, and also in the preparation of certain painters' colours. The iron, freed from tin, is washed in water, then in weak alkali, and, finally, again in water. It is sold to iron-works, where it is introduced in the puddling process.

The tinplate waste is brought into a perforated copper cylinder, which is brought by a crane to a series of vats placed in a semicircle round it. All the vats are provided with bearings for the axle of the cylinder, which is then rotated by mechanical power. The cylinder is 1·8 metre (71 in.) long, 1·95 metre (78 in.) in diameter; the body is made of sheet-copper, 3 mm. (0·12 in.) thick, whilst the ends are 7 mm. (0·28 in.) thick; the body is provided with holes 9½ mm. (0·38 in.) in diameter, and 50 mm. (2 in.) apart, it is bound round with copper rods 10 mm. (0·4 in.) in diameter. A door with two flaps serves for the introduction and removal of the tinplate. This drum is brought in succession into each of the four vats mentioned above, of which the first is the acid bath, the second is a slight alkaline bath, the third water, and the fourth again an alkaline bath. The four vats are of

equal size; they are made of fir, 64 mm. (2·5 in.) thick, are 1·3 metre (52 in.) deep, 2·2 metres (86 in.) long, and 1·9 metre (75 in.) wide. The bearings for the drum are so placed in all the vats that it is only half immersed. The first vat, which contains the acid bath, is lined with sheets of glass, cemented together with a mixture of sulphur and steatite. The charge for the cylinder is 430 kilos. (967 lb.), and the duration of the operation, including emptying the drum, seventy-one minutes. For 1000 kilos. of tinplate, 242 kilos. of hydrochloric acid and 7 kilos. of nitric acid are required on the average. The nitric acid is added to the hydrochloric acid when the latter is almost exhausted. At the commencement of the process, the acid vessel is two-thirds filled with hydrochloric acid of 20° B. After complete saturation, the acid is evaporated in copper pans to one-third of its volume, any lead which may be present precipitated by sulphuric acid, the clear solution drawn off into a large wooden tub after the lead sulphate has settled, and, after dilution with double its volume of water, the tin precipitated by zinc; 32·6 parts of zinc are required to 58 parts of tin.

According to Ott, the process of Seely (*Muster-Zeit.*) is much used in Switzerland, and with great success. The apparatus employed is a cylinder of boiler-plate, the bottom and top of which can be removed and closed by a glycerine lute. A pipe enters the upper part of the cylinder, also through a glycerine lute. A delivery-pipe leads from the lowest point of the bottom. In the lower part of the cylinder there is a perforated double bottom, which can be placed parallel with the bottom, or turned vertically down. The tinplate is heaped up on the false bottom, the bottom and the cover attached, and then chlorine gas introduced through the delivery-pipe. The reaction begins at once, and continues for a longer or shorter time according to the quantity of the tin scrap; about five to six hours are required to remove the tin from 10 cwt. The resulting gaseous tin chloride may be passed directly into water; it is quite free from iron. To empty the apparatus, the bottom is removed, and then the false bottom, with the remains of the tinplate, allowed to fall down. The average quantity of tin contained in waste

tinplate is 5 per cent., thus, from 1000 lb., 111 lb. of anhydrous stannic chloride and 950 lb. of iron are obtained, for which 80 lb. of chlorine are sufficient.

Another method for utilising copper and tinplate waste consists of the following process (*Dingler's Journ.*, 219, p. 96):—The waste tinned iron is introduced into copper solutions, in which the copper is present as chloride or sulphate; if this is not the case, sodium chloride and sulphate are added. The tin is loosened and precipitated as hydrate, the bared iron then precipitates the copper from solution. The stannic hydroxide is removed before the reducing action of the iron commences.

Noteworthy and very practical observations in regard to the treatment of waste tinplate have been made by C. Künzel of Blasewitz, near Dresden (*Berg- und hüttenmännische Zeit.*). The purchase of tinplate waste requires a certain amount of caution. The thinner the tinplate, the more tin it contains. French tinplate contains more tin than English. Very frequently, however, a mixture of tin and lead is used for tinning iron; if the lead exceeds 10 per cent. of the tin, the tinplate should be avoided in buying; such tinplate must always be separately treated, since it offers great difficulties in the process. Lacquered tinplate is also to be avoided, since the lacquer is not thoroughly attacked by boiling in dilute acid; it is therefore necessary to destroy it by a gentle preliminary heating, which causes the yield of tin to be somewhat lower. In delivery contracts with dealers the quality of the tinplate must be exactly stipulated. Since it is impossible to stipulate for a certain proportion of tin in buying tinplate waste, the material must be inspected, a price offered according to the thickness of the plate—the thinner the more valuable—with the arrangement that, if iron coated with lead or zinc is added, the delivery will be rejected. When old barrels or packing-cases are to be had cheap, the waste is stamped down in them for transport; otherwise, compressed packages are made, weighing 1–2 cwt., by stamping the waste into a strong rectangular wooden box, somewhat wider at the top than the bottom, by means of a strong rammer, and then binding the packages together by means of two crossed iron wires or old hoop iron.

In treating these compressed packages of waste tinplate, it is necessary to loosen them as far as possible, so that two surfaces of tin may not be in such firm contact that the acid cannot penetrate between them. This is best done at first with a large three or four-pronged fork, and afterwards by loosening and bending by hand.

To dissolve the tin from the waste tinplate Künzel employs a boiling mixture of one part of strong nitric acid and ten parts of strong hydrochloric acid diluted with so much water that the liquid remains about four inches deep over the waste when the boiling is finished. The liquid is contained in wood or brick vats lined inside with a mixture of two parts of sand and one part of sulphur, applied hot. A pipe of vulcanised rubber reaches to the bottom of one of these vessels, which should not have a less capacity than 1 cub. metre (35·3 cub. ft.); the vulcanised pipe is fastened above to a copper pipe, which is in communication with a steam generator.

The empty vat is almost completely filled with the loosened tinplate waste, then the acid mixture is added after it has been diluted to such an extent that it covers about four-fifths of the tinplate. Steam is now allowed to enter in order to heat the solution to boiling; the introduction of steam is continued, with the valve only so far opened that the liquid just boils, until the upper parts of the tinplate are entirely freed from tin and the evolution of hydrogen has almost stopped. On an average, the boiling lasts thirty to forty-five minutes. The solution, which contains all the tin and a certain quantity of ferric chloride, is run off whilst still hot, by opening a tap at the bottom of the vessel, into a tank, where the greater part of the lead chloride separates on cooling. On the average, for 1000 kilos. (1 ton) of tinplate containing 5–6 per cent. of tin, there are required 300 kilos. (6 cwt.) of hydrochloric acid and 30 kilos. (66 lb.) of nitric acid, which are diluted with about 3·5–4 cub. metres (123–140 cub. ft.) of water or wash waters.

The residual iron is washed with water in the dissolving tank, the washings being used to dilute the acid for the next operation. The iron is then lifted out by means of forks, and

as quickly as possible stamped into bundles. It is dangerous to keep this iron waste piled up loosely in large heaps, since it readily oxidises, and may become red hot. For the treatment of 3000 kilos. (3 tons) of waste tinplate per day of twelve hours, six or seven dissolving tanks are required, each of 3 cub. metres (106 cub. ft.) capacity, otherwise an excessive amount of labour is requisite.

The cooled solution drawn off from the tinplate is brought into large wooden tubs or stone tanks filled with old zinc plates, old zinc roofing sheets, etc., by which the tin is precipitated, together with any lead present in the liquid. The precipitation must take place without any noteworthy evolution of gas; if any effervescence occurs, the solution is too acid and zinc is wasted. The filtered solution is tested by slightly acidifying, and passing in sulphuretted hydrogen, to see if all the tin is precipitated. The precipitation requires two hours at the most. A tap at the bottom of the tub is then opened, and the clear solution, which it is impracticable to utilise further, run off through a strainer of sailcloth. The zinc remaining in the tub is then gently shaken to remove the deposited spongy tin; the tub is again filled with the solution obtained by boiling the tinplate, and the process thus continued, with occasional replacement of the dissolved zinc, until the tub is filled to the extent of one-third to a half with spongy tin.

The spongy tin is removed from the tub and washed through a metal sieve with a mesh of 3–4 sq. mm. into a sailcloth strainer by means of a stream of water. Small undissolved pieces of zinc remain on the sieve and are returned to the precipitating tub. Solder is also caught by the sieve; it is melted and sold as soft solder. The spongy tin which has passed through the sieve is washed with water on the linen filter so long as the washings contain iron. It is then squeezed in linen bags under a hydraulic or screw press in order to remove as much water as possible. The spongy tin so obtained is now converted into tin crystals (stannous chloride); it is desirable to dissolve it in hydrochloric acid immediately after it has been pressed. The manufacture of tin crystals is sufficiently well known; Künzel

therefore gives further only the treatment of the insoluble residue obtained in dissolving the spongy tin. This consists principally of lead chloride and stannic oxide; Künzel has successfully treated it by mixing with about two volumes of common small coal, and heating it to a red heat in a small Belgian zinc furnace, with six retorts set in two series at a considerable inclination to the front. If sufficient lead chloride is not present, it is added from that obtained in the cooling-tanks from the tin solution. All the tin present then passes into the receiver as stannic chloride, whilst metallic lead is formed at the same time; the latter partly runs into the receiver, and is partly recovered in grains by washing the residue in the retort.

In regard to the treatment of the residue of iron, Künzel remarks that, with a daily treatment of a small quantity of waste tinplate, and where sulphuric acid is to be obtained at a low price, the waste iron may be profitably converted into green vitriol.

Künzel obtained a good product by making the waste iron up into bundles of about 5 kilos. (11 lb.) and introducing it into the puddling furnace at the moment when the iron is in the most spongy condition, the quantity being 10–20 parts to 100 parts of pig-iron. An excellent white cast-iron was obtained by melting 2 parts of the waste iron with 5 parts of grey cast-iron turnings in the cupola furnace.

W. D. Walbridge proposes to immerse waste tinplate as the positive pole in a bath containing 3 kilos. of caustic soda, 1 kilo. of sodium nitrate, and 7 litres of water; or 3 kilos. of caustic potash, 1 kilo. of potassium nitrate, and 7 litres of water; or, finally, 0·2 kilo. of caustic potash, 2 kilos. of common salt, and 7 litres of water, whilst the iron vessel forms the negative pole. The inventor has obtained a patent for the process.

E. Roussett recommended the following method:—In the first place, the tin must be heated in an oxidising flame, which oxidises all the free tin, and also that combined with iron. The tinplate is now covered with a brown, brittle crust, the upper layer of which consists of stannic oxide, and the lower of the magnetic oxide of iron; it is then squeezed between

grooved rollers in order to remove the oxides, which are sieved and collected. The residual iron gives a good wrought- or cast-iron, but is said to be especially suitable for precipitating copper. The stannic oxide, although mixed with the iron oxide, can easily be reduced to metallic tin by the usual methods; the metal obtained is free from sulphur and arsenic.

CHAPTER LVII

WOOL WASTE

Utilisation of Waste Wool in making Shoddy, etc. (Grothe, *Technologie der Gespinnstfasern*, vol. i., p. 209 *et seq.*; Muspratt, *Prakt. Chemie*, vol vi.).—For this purpose rags and all waste containing woollen fibre, thus also mixed fabrics of cotton and wool, are employed. The rags and waste are first subjected to certain preliminary operations in order to remove buttons, seams, string, etc., when about 20 per cent. of waste is produced, which is used in agriculture. The rags are then cut into pieces of suitable size, and sorted, first into pure woollen and mixed fabrics, then according to the character of the wool fibres, whether short or long. The short-fibred rags consist mainly of cloth and all materials of carded yarn, milled or not; the long-fibred consist of all unmilled, and especially of worsted, fabrics. The names of mungo and shoddy are used respectively for the materials differentiated in the last sentence, after they have been torn up. Finally, there is a selection according to colour.

In the following we proceed exactly according to the authorities quoted. Since rags come into commerce unsorted, or only superficially sorted, the manufacturer has to undertake the operation. This, however, loosens the dust in the rags, and the air of the sorting-room is soon something more than saturated with it; good ventilation is accordingly requisite, or the rags must first be cleaned. Breton, of Pont de Claix, accomplishes this purpose by arranging the rags in a layer 30 cm. (1 ft.) deep and moistening with bleaching-powder solution, 0·5 litre per sq. metre (1 gal. per 100 sq. ft.). They are then brought into a species of corn-sifter provided with a fan, which drives out the dust into a flue 5–6 metres (16–20 ft.) long, where it deposits. At the outlet is a

water-pipe which gives a fine spray of water and so throws down all the dust. Especially dirty rags are boiled with milk of lime and a little soda, washed in a washing-machine, and

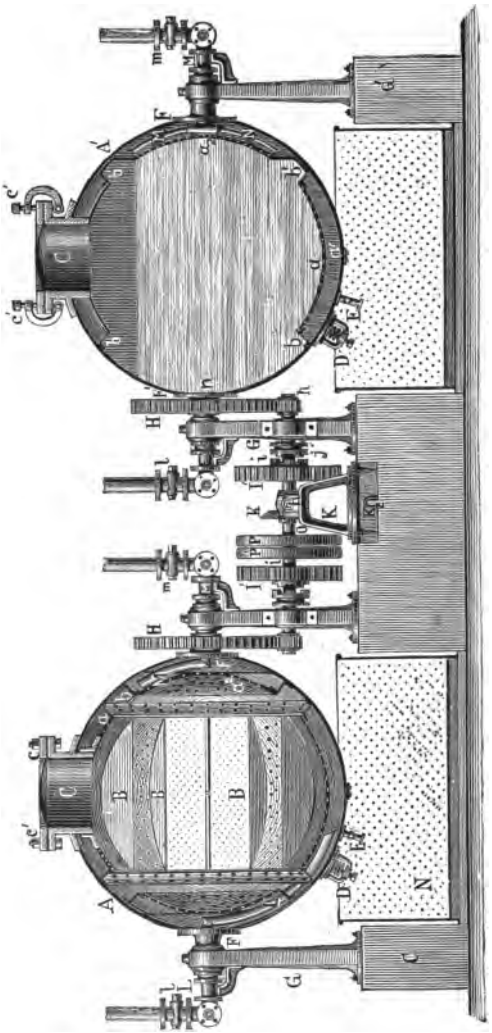

FIG. 12.

then dried. A rag-washer, which works very well, has been constructed by Planche & Rieter (*Polyt. Centralbl.*). This machine, which is made by Seraphin, of Paris, has two

spherical vessels for the reception of the rags; the two may be worked together or separately, for which purpose the driving mechanism is provided with the requisite clutches.

Fig. 12 is a vertical section through the axes of both vessels, Fig. 13 a ground plan, and Fig. 14 an elevation, of the driving mechanism. In Fig. 12 are shown the different systems used for introducing the steam and the washing liquid, the various possible modifications being represented at the same time.

Each of the spherical vessels A and A^1 is about 6 ft. in diameter, can contain about 1300 lb. of rags, and is con-

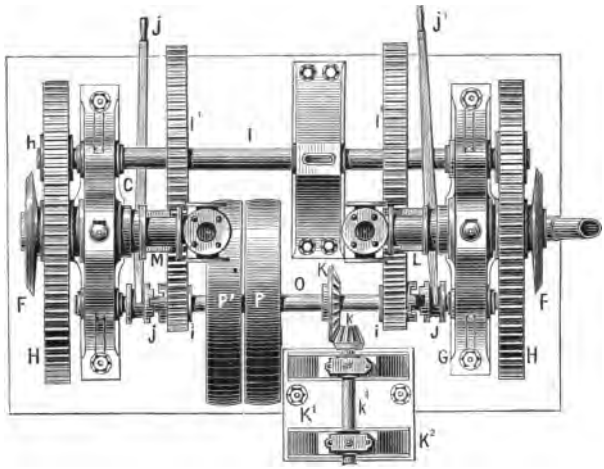

FIG. 13.

structed of $\frac{3}{8}$-inch boiler-plate, so that it can resist a steam pressure of five atmospheres. Each also contains a double perforated bottom of thinner plate a, fastened to the outer walls by angle irons b. The holes in these double bottoms are $\frac{5}{16}$ in. wide, and are at distances of 1 in. apart. Over this bottom are scrapers B or rods 18 in. long, which separate the rags and keep them in motion. The steam enters the ring-shaped space a' between the perforated bottom and the outer wall. The manhole C attached to the vessel serves for the admission of the rags and in case of repairs; it is closed by a cover fastened down either by ordinary bolts as shown for the vessel

on the left, or, as seen on the vessel at the right and in Fig. 15, by a swinging nut c' moving on a hinge. The valve D, shown magnified and in section in Fig. 16, is also affixed to the exterior of the vessels; it serves as a safety-valve against too strong pressures, and can also admit air in case of the production of a vacuum. It has a brass seating, fitting on to the spherical vessel, in which moves the real safety-valve d, provided with a conical face tapering downwards and pressed down by a strong spring e, which works against the plate f of the arch D, which again carries a screw f' for regulating the pressure of the spring. The valve d is again in its turn perforated and carries the second valve d', opening inwards, which is kept pressed into its seat by the weak spring e.

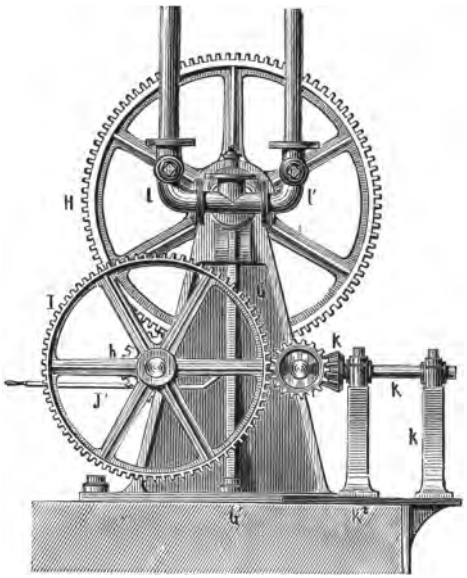

Fig. 14.

A tap E on each vessel, opposite to the manhole, serves to run off the liquor or washing liquid. Each vessel has two pinions F and F', which are carried by the columns G bolted to the brickwork G'. The longer axis F' carries a large cog-wheel H, which is actuated by the cog-wheel h. Both the wheels h are on the shaft J, which has its bearings in the columns G and in a third and lower one. The main shaft O, with the pulleys P and P', moves the shafts I, etc., by means of the cog-wheels i and i'. In order that each vessel may be revolved separately, the cog-wheels i are not attached to the shaft O, but are made to rotate by means of the clutches j and j', worked by the levers J and J'. Further, in order to

be able to move the vessels by hand so that the manhole may be exactly at the highest or lowest point, in order that the rags can be introduced or removed, the conical cog-wheel k is also attached to the shaft O. It is moved from the subsidiary shaft k', to which a handle can be attached, and which is supported on bearings in the supports k^2.

From Fig. 12 it is seen that the steam and the various washing liquids can be conducted through the hollow axes F and F' in various ways. On the left side of Fig. 12 there is a central pipe L which rests in a small support and divides into two branches, provided with the taps l and l' (Figs. 12 and 14), in order to be able to cut off the pipes. In the interior of the vessel, is seen the pipe L continued downwards by the pipe L', which terminates between the outer wall and the perforated bottom and rotates with the vessel. The fixed

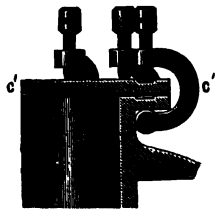

FIG. 15.

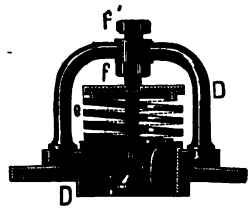

FIG. 16.

pipe M enters through the other axle F', it also divides outside into two branches with taps m and m', whilst the other end is continued inside by the arm M, which is screwed on in an upward direction. A perforated diaphragm a' at the side protects the pipe from contact with the rags. The steam enters through this pipe at a pressure which can be regulated as required.

The pipes affixed to the vessel on the right are differently arranged. The steam there enters on the left through a rose n. On the opposite side are to be seen two fixed pipes M and N, of which M is directed upwards and introduces the steam, N downwards and introduces or removes the washing liquid. Both these pipes are connected by a T-piece with the central pipe M which passes through the axle F,

and is separated by a diaphragm into two divisions. The inside pipes *M* and *N* are here also protected from the rags by a perforated wall almost concentric with the spherical vessel and somewhat differently fixed. A tank *N* perforated by many small holes is placed under each vessel. It may be set on wheels so that it can readily be moved on a tramway. It is intended to receive and drain the washed rags which fall out of the emptied boiler.

These machines work exactly like the ordinary cylindrical form. When in the position of Fig. 12 they can be filled through the manhole, which is then closed tightly. When

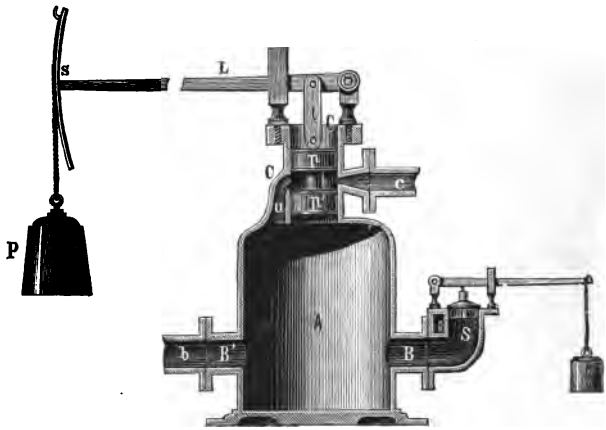

FIG. 17.

the washing is finished, the liquid is first run off by the tap *E*, then the sphere is turned so that the manhole is at the bottom. The latter is then opened, and the rags fall out.

It is necessary to produce a constant uniform steam pressure, which is effected by means of the regulator, shown separately in Fig. 17. It consists of a vessel *A* with a cylinder *C* cast on at the top, and with two side pipes *B* and *B'* placed opposite to one another, of which *B'* is used to carry away the steam, whilst a safety-valve *S* is screwed on *B*. The cylinder *C* is provided with a bored metallic lining, in which works the double piston pp^1. Between the two discs of the piston communication is made with the vessel *A* by means

of the side passage *a*, the opening of which is opposite to the pipe *c*. The top of the piston is connected by the rod *l* with the lever *L*, on the end of which is an arc *s* to carry the weight *P*. When, for example, the safety-valve is loaded to four atmospheres pressure, which is not to be exceeded, the weight of the lever *L* must be so regulated that the space between the two discs of the piston just makes communication between the channel *a* and the entry pipe *c*. If now, *e.g.*, steam of six atmospheres enters, the piston rises, and the openings of *c* and *a* are narrowed so that little steam can enter the vessel *A*. The safety-valve *S* prevents any accidental accumulation of pressure.

According to the mode of operation there are four different methods of manufacture for mungo and shoddy (following Grothe's account):—

1. The woollen rags, unwashed and only moistened, are torn up, and are not further treated in the carding-machine, but are at once put on the market. The loosened mass thus still contains all the dust and dirt.

2. The woollen rags are not washed before tearing, but are afterwards treated dry in the carding-machine, by which a quantity of dust is removed.

3. The woollen rags are not washed before tearing, but are afterwards somewhat oiled and carded. This system affords the greatest opportunities for fraud. In the first place, the oil fastens the dust on the fibres, and also those fibres which are too short for further treatment, and almost like dust, to the longer fibres. When this carded mungo comes into the market it generally deceives the purchaser. It is different when the mungo manufacturer spins it himself.

4. The woollen rags are well washed before tearing, and afterwards are not oiled or carded. This method always produces a very clean and unmixed fibre, free from all impurities. It also avoids the production of dust during the treatment, which injuriously affects the workpeople in the other methods.

The process of the treatment has now been sketched. After freeing from buttons and seams, the rags are cut into small pieces and then go to the "devil," which tears

them into fibres. This procedure is adopted in methods 1, 2, and 3; but in method 4 the rags are washed before tearing, and again afterwards, then centrifugalised and dried. The "devil," or tearing-machine, consists of a large drum, the surface of which is thickly studded with iron teeth, and which moves with a great velocity—about 550–700 revolutions per minute. The rags are fed to the drum between two rollers of small diameter provided with longitudinal grooves. These rollers revolve very slowly and, since the upper is pressed very hard on the lower by means of a lever and weight, the rags are held fast and the teeth of the drum comb out only the cross threads. This operation produces a fibrous mass, which is termed mungo when it originates in cloth and carded yarn fabrics, and shoddy when it is produced from worsted and long-fibred materials.

In the above methods a treatment in the willy follows on the tearing by the "devil." The rollers of the former have strong wire points with an elastic attachment to the surface. Mungo is generally not similarly treated in the willy and the following carding-machine, but is at once packed and afterwards carded mixed with natural wool in proper proportions, since it is too short in the fibre to be spun alone. The quantity of mungo varies from 20–70 per cent. to 80–30 per cent. of natural wool. The subsequent spinning offers no further difficulties. Shoddy wool is at once carded on the willy and the following machines, and is generally spun without further additions.

There is a great quantity of rags composed of mixed fibres. Generally the warp is of vegetable fibre and the weft of animal fibre. For mungo-spinning, the vegetable fibre must be removed. Generally the two textile fibres are separated by treating the rags with acids, which destroy the vegetable fibres, but do not attack the wool. Sulphuric acid diluted to about 18° B. is used, and also hydrochloric acid. After the acid bath, the material is brought into alkaline solutions, in order to neutralise the acid remaining in the fibre; it is then well dried. In consequence of this treatment, the vegetable fibre has been converted into a friable condition, whilst the wool fibres remain uninjured, and can

be isolated simply by rubbing. For this operation, many different methods are now employed, which will be mentioned below.

Newmann separates the wool from mixed woollen fabrics as follows:—In order to protect the wool from the action of the acid, it is saturated with a solution of sulphate of alumina or alum (1–5 parts to 100 parts of water), and then dipped in a warm soap solution containing 1·5–7·5 parts in 100 parts of water. The material to be treated, after this preparation, is brought into sulphuric acid (1–5 parts diluted with water to 100 parts). The moist fabric is then subjected to a temperature of 95° C. The vegetable fibre is then so thoroughly destroyed that it can be removed by rubbing or washing in hot water; the wool fibre, however, remains unimpaired.

R. Böttger omits the preliminary treatment with alum, and applies a soda bath after the treatment of the fabric, in order to neutralise the acid. The carbonic acid, which is then evolved, considerably loosens the residual wool.

Ward gives a process for separating cotton from wool, which has for its object the recovery of the cotton for paper-making. He subjects the mixed fabric to a pressure of from three to four atmospheres in a Papin's digester. The cotton then remains quite unaltered, whilst the wool is converted into a friable substance.

In order to recover the woollen fibre from a fabric composed of wool and vegetable fibre, Schaller immerses it for twelve hours in a bath composed of 3 per cent. of sulphuric acid (66° B.) and 97 per cent. of water, and then dries. If this solution has not had sufficient action, the rags are then exposed for from four to five hours to a temperature of 60–70° C., when the vegetable fibre becomes friable.

Rowley recovers the wool from mixtures with vegetable fibres by treating the rags with dilute sulphuric acid, then bringing them into a rotating wire sieve, and there drying them by a current of hot air. The rags are then packed in boxes with sand, where they remain for a longer or shorter time, according to their nature, when they are again separated from the sand by the rotatory action of the wire sieve. This

method is so complicated, and its effective action appears so questionable, that it can in no way be recommended.

Grothe calls attention to the fact that the greatest care must be given to the strength of the acid bath, in order that the wool hairs may not be attacked.

Extracted mungo wool is mixed with mungo obtained by the mechanical methods, and the two worked up together.

A. Gawalowski, of Brünn, has obtained a patent for a process for the recovery of the **fat from wool-scouring waters,** and other industrial waste waters containing soap. The essential features of this process are: sulphurisation of the suds and acidification; the sulphuretted hydrogen, at the moment of its production, acts on the resulting scum, destroying the aniline dyes to a great extent. The decanted scum is impregnated with chromates, by which the size, starch, and dextrine are partially oxidised; this process takes place slowly, but is more complete on standing. The scum, after this treatment, is washed, by which the dyes, the oxidised mineral pigments, and also the glutin and oxalate products are removed. The wash water is deep red to brown. At the same time, the scum is already oily, can be kneaded in water like butter or lard, but can no longer be mixed up with it to a liquid similar to the original suds, as can the fresh scum. It still contains excess of chromates and traces of free acid, and would therefore easily destroy press cloth. Gawalowski accordingly stirs it with a reducing solution, which changes the chromate to chromium oxide, and then presses out the neutral green liquid in the cold, after which the cake of scum is pressed warm. By this method of treatment there is obtained the highest yield of fat, and a more pure product, which can well be used, after the acid has been removed, in spinning dark coarse yarn, and also in soap-making.

Manufacture of Yellow Prussiate of Potash from Wool (horn and blood).—J. J. Hess, of Vienna, has given a long account of his own experience of this manufacture (*Deutsche Gewerbe-Zeit.*). The proper preliminary treatment is exceedingly important to lucrative working, it consists in a cautious drying at a high temperature, or rather in gently calcining the nitrogenous material, in order, in

the first place, to drive out all moisture and, in the second, to make the nitrogenous matter pulverisable and also richer in nitrogen. This process of concentration is best conducted in a kind of "fig-coffee" drum, or in a large rotatory apparatus, capable of being heated. One axle must be hollow, and be connected, by means of a bent pipe, with a cooling tank, which condenses the vapours evolved, since these are also valuable, consisting of combustible oils and ammonium carbonate.

The dried, roasted, and concentrated mass, when the operation has succeeded, must be frothy, brown, transparent, interspersed with bubbles, of a slight, peculiar odour, and very easily friable. If too strongly burnt, the mass appears black, is very light, very frothy, and very poor in nitrogen; on this account every attention must be given to the drying process, in order that heavy loss may not result.

The next most important material is the potash, which is frequently, through false economy, bought of bad quality, and thus made an inconvenience in the manufacture. The potash should be as pure as possible.

Next comes the third raw material, the iron, which can be used in the form of hammer scale, dull-coloured ferric oxide, or iron filings and turnings, in fine powder.

All the substances are to be made and kept quite dry, and in a state of fine powder; they are then mixed for use in the following proportions:—10 kilos. of dried horn, etc., 6–8 kilos. of potash, 4 kilos. of hammer scale, etc. The average produce is 3–3½ kilos. of yellow prussiate.

The intimate mixture is now heated in clay crucibles lined with sheet-iron, in a muffle furnace very similar to an ultramarine furnace. After about twenty-four hours the furnace is opened, the crucible removed, and the coherent mass, which readily falls out, thrown at once into cold water in order to prevent oxidation. As soon as the mass falls into the water a hissing is heard, and frequently sparks momentarily fly out, which is a sign of a successful operation. The mass soon falls to a fine black powder, leaving a clear, pale-yellow solution, which becomes dull in colour when subsequently boiled, and deposits fine crystals.

In addition to other advantages, Hess's process also utilises the resulting fine powder. The black deposit contains pure iron and carbon; the iron can be extracted by nitric acid and used in the preparation of Prussian blue, or it can be transformed into ferric oxide by burning in the air, and used over again. The nitrate of iron, which is thus obtained, may be acidified and used to act on the mother liquors, when Prussian blue and potassium nitrate are obtained. The carbon left in very fine powder, after the iron has been dissolved out, gives an excellent pigment; after washing and grinding, it has great covering power and a fine, pure shade.

For the **treatment of the suds of wool-washing and cloth fulling**, E. Neumann, of Rosswein, has obtained a patent (*Neueste Erfindungen und Erfahrungen*). In either case the whole apparatus consists of two tanks dug in the ground (even for large factories two oil-vats of 12–15 cwt. capacity are sufficient), to which the suds are run by sluices or pipes from the washing-machines or cylinders, and also of a lift and force-pump, and a filter-press with waste channel.

Crude calcium chloride (or some substitute, which forms, with fats, soaps insoluble in water) is dissolved in an equal weight of water and the solution, to the extent of 1, 1·5, or 2 per cent. of the total capacity, according to the amount of fat in the suds, poured into tank No. 1. The suds are then run in; the ensuing commotion ensures a thorough admixture with the calcium chloride solution; the lime soaps are at once formed and separate in flocks, which remain suspended in the water. When the first tank is full, the contents are pumped into the filter-press, whilst in the meanwhile the suds are being run into the second tank, which has just previously been charged with the necessary amount of calcium chloride.

In the filter-press remain lime soaps, dirt, hair, etc., whilst the clear water, carrying with it chiefly potassium and sodium salts and a little excess of calcium chloride in solution, runs away by the waste-pipe. After tank No. 1 is emptied, tank No. 2 is pumped out, No. 1 again filled, and so on, until after about eight to twelve hours' work the press is full, a condition readily recognised when the taps only drip slightly. The

press is opened, and the cakes, when they do not fall out unassisted, removed by a wooden spatula. The press is then closed, and the pump again started, when the process begins afresh. Only the introduction of the calcium chloride, the opening and closing of the feed-cocks, the removal of the press-cakes, and the starting of the pump, require manual attention; the whole is the work of but a few minutes, consequently a special workman is not requisite.

The press-cakes obtained are either dried in the air and used to prepare an oil-gas, or are, more profitably, treated to recover the fat, which is a separate part of the treatment of the suds. It may be undertaken by large mills and wool-washing establishments; smaller works find it more advantageous to sell the fat-cakes to the grease-works.

In recovering the fat, the press-cakes are stirred with very dilute hydrochloric acid to a thin paste; if necessary, more hydrochloric acid is added to produce a weak acid reaction. The mass is then forced through a filter-press heated by steam. The fat and the re-formed calcium chloride run into small vats, from which, after some time, the calcium chloride solution, containing excess of hydrochloric acid, is drawn off into a vat containing carbonate of lime, in combination with which the hydrochloric acid again produces calcium chloride, which is used to produce fresh quantities of lime soaps. Thus it is only requisite to procure a supply of calcium chloride once for all. The fat is heated in a pan over an open fire to 70° C. with 10 per cent. of sulphuric acid of 10° B.; after standing some time it forms a clear layer on the top, which is skimmed off into barrels, and is then ready for sale.

If, instead of calcium chloride, chemicals be used which it is not necessary to regain on account of their low value, the recovery of the fat is still simpler and cheaper.

Cotton-Spinners' Waste in the Manufacture of Guncotton.—According to the English patent of Mackie, the waste of cotton-spinning is treated with superheated steam in order to remove the oil, then carded and immersed in the usual acid mixture—300 parts of a mixture of 1 part of nitric acid (specific gravity, 1·52) and 3 parts of sulphuric acid (1·85), cooled to 5–25° C. The guncotton produced is carefully

washed, cut to pieces by means of a rapidly rotating circular knife, and finally passed between rollers, from which it issues in a very finely divided state. This cutting and squeezing are said to have the advantage over the usual pulping in the beating-machine, that all the capillary tubes are completely destroyed, and thus any residual acid can readily be washed out.

The succeeding operations consist in mixing the nitrated cotton with saltpetre and sugar (67 parts of guncotton, 28 parts of sugar, and 5 parts of saltpetre), granulating the mixture by means of wire sieves, and drying in tightly closed pans heated to about 38° C., in which a vacuum is maintained by a pump. An increased addition of sugar and saltpetre decreases the violence of the explosion of the guncotton.

According to the English patent of J. Hall, rags and other linen waste are brought into a bath composed of equal volumes of nitric acid (specific gravity, 1·5) and sulphuric acid (specific gravity, 1·84), and diligently stirred in it for twelve to twenty-four hours. The mass is then allowed to drain, pressed, carefully washed for seven to fourteen days, and dried. For purposes for which a violent explosion is not required, the guncotton, whilst still moist, is mixed with starch paste.

Heddebault discovered a method by which wool can be removed in the dissolved state from fabrics in which it is contained mixed with cotton or other vegetable fibres. When these fabrics are subjected to the action of superheated steam under a pressure of five atmospheres, the wool dissolves, and falls to the bottom of the vessel, whilst the cotton, flax, or other plant fibre resists the action and remains in a condition suitable for the manufacture of paper. The fluid mass, in which the dissolved wool is contained, is evaporated to dryness; it is completely soluble in water. Rags treated in this manner gain in value to an extent sufficient to cover the cost of the process.

In conclusion, it is known that carpets and rugs can be made from various rags; they are constructed by sewing together small patches, cut uniformly, of various sizes. A better method is to cut the rags into narrow strips about 0·5

cm. ($\frac{1}{4}$ in.) wide. Felt is cut in a circle in order to obtain very long strips; other rags are cut lengthways. The strips are sewn together, and wound into balls, which are given over to the linen-weaver. When the rags are sorted as far as possible according to colour and material (whether linen, wool, jute, or cotton), pretty striped carpets are obtained, which are cheap and wear well.

CHAPTER LVIII

WOOL SWEAT

Potash from Wool Sweat.—According to the patent of Prof. K. Kraut, of Hanover, wool is washed in the ordinary washing-machine with warm water, to which potash is added. The wash waters are allowed to settle in tanks, the residual liquid evaporated to dryness, and heated on the hearth of a reverberatory furnace. The residue contains the potash used in washing, and the potassium which was present in the sweat, also mainly in the form of potash (carbonate). A portion of the potash solution obtained by lixiviating this residue is again used for washing wool, whilst the remainder may be evaporated for sale, and the residue calcined.

Treatment of Wool-Washing Waters with Baryta.—Daudenart and Verbert, of Schaerbeck, near Brussels (*Deutsche Industrie-Zeit.*), mix a solution of barium chloride in a closed vessel with magnesium carbonate, whilst carbon dioxide is at the same time introduced; barium carbonate is then obtained. The magnesium chloride, which remains in solution, is converted by superheated steam into hydrated magnesia, when hydrochloric acid is obtained as a by-product. This process is designed for the utilisation of wool-washing waters, which are mixed with baryta solution, the precipitate pressed and decomposed with hot dilute hydrochloric acid. The separated fat is skimmed off, the liquid evaporated, and the residue of barium chloride converted into barium carbonate by the method given above.

The liquid from which the barium soap has been removed is evaporated to dryness, and the residue, consisting mainly of potash, calcined, and converted into refined potassium carbonate in the usual manner.

CHAPTER LIX

THE WASTE LIQUIDS FROM SUGAR-WORKS

Utilisation of the Waste Water.—W. Riehn has described a process (*Dingler's Polyt. Journ.*, 223, p. 402) by means of which it is possible to remove the injurious effects of the waste waters, and to recover them again with little loss, not, indeed, in the form of a potable water, but in such a condition that they give no trouble in the neighbourhood, and are quite suitable to be used over again in the works or elsewhere. In using this process, in order to avoid a false impression of the extent of the purification it affords, it must not be neglected to remove entirely the deposits in the drains and other legacies of the past. The process may be carried out without any considerable outlay, which is covered by the value of the fertiliser obtained, and by the labour of one man, under careful supervision. The purification is said to attain three objects:—prevention of conditions dangerous to health, production of a valuable fertiliser, and recovery of the water. In regard to the operations to be performed, the impure waste waters may be divided into—(1) water used for washing the beet, and from the crude juice station; (2) water from the bone-black treatment, and from washing filters and cloths; and (3) condensations and condensed water from boiling the juice.

The condensed water from the steam-engines and other apparatus, which is collected separately and carefully, the water from the evaporators, and the waste juice from the filters, do not need special treatment here; they are already employed to the best advantage in any properly conducted works—for feeding the boilers, treating the bone-black, slaking lime, washing — and give rise to no further inconveniences.

1. Water Used for Washing the Beet, and from the Crude Juice Station.—The temperature of these liquids in the waste pipes varies between 18° and 32°C.; their impurities are largely earth, mechanically carried, other dirt and particles of beet, also juice in small quantity, *i.e.* sugar, salts, etc. The purification of these waters is very simple; the

FIG. 18.

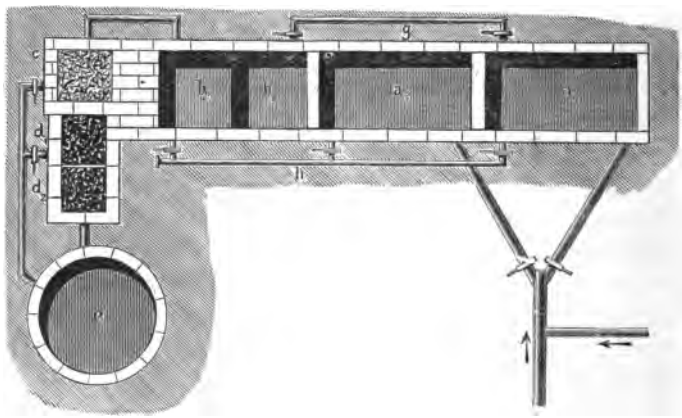

FIG. 19.

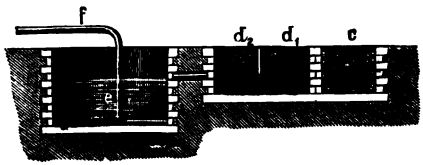

FIG. 20.

settling-tanks a_1 and a_2 (Figs. 18–20) are quite sufficient for the impurities just mentioned; the small quantities of sugar, salts, etc., are also completely removed by the contents of the filters c, d_1, and d_2, after the liquid has passed through the tanks and undergone the reactions which take place there, as described under the next section (2). The water received in the collecting-tank e is at least as pure as the original water

used, and the necessary cooling has been obtained in the open tanks and conduits. The settling or mud-tanks, a_1 and a_2, are of masonry, more or less sunk in the ground according to the local conditions; for the daily treatment of 100 tons of beet, each is 8·8–9·5 metres (29–31 ft.) long, 3·8–4·4 metres (12·5–14·5 ft.) wide, and 1·9 metre (6·2 ft.) deep. In the case of larger quantities, or very dirty beet, a third such tank should be provided. The tanks b_1 and b_2, also of masonry, are together as large as a_1 or a_2: they are merely divided by an overflow partition. It is necessary that the valves shown in the figures should be properly arranged, and that all the pipes should have the correct fall. The manipulation is simple: at the commencement, all the waste waters run to a_1, thence through the pipe g to the tank b_1, etc. When a_1 is filled with deposit, the valves are transposed; the waste waters then go to a_2, whilst a_1 is emptied. From the tanks b_1 or b_2, in which the last settlings are deposited, the liquid comes to the first filter c, which is 1·9–2·5 metres (6·2–8·2 ft.) square or in diameter, and is the same depth as the previous tanks; it is filled with layers of various grades of cinders, sand, or other material suitable for the complete mechanical purification of the water. The filling material may be suitably mixed with alum mud, iron chloride, potassium permanganate, etc., in case such substances are to be obtained cheap in the neighbourhood of the work. From the filter c the water enters the filter d_1 at the bottom, from which it flows over into d_2 (1·6–1·9 metres square or in diameter, *i.e.* 5·3–6·2 ft.), and finally through a ground pipe into the collecting-tank e (3·2–3·8 metres in diameter, *i.e.* 10·5–12·5 ft.), whence the purified water is removed to the works by the suction-pipe f.

The contents of the filter are to be changed as soon as it is so choked that it no longer works. Turf coke is the most suitable filling for d_1 and d_2; when it cannot be obtained, waste bone-black is used, or the prepared wood charcoal of Jünemann—charcoal in coarse grains, boiled with a solution of five parts of acid calcium phosphate and five parts of sulphate of alumina, then dried and ignited. This charge is to be renewed during the season only once, or several times at the most, as soon as the diminished purity of the filtered water

makes it necessary. The pipes must be laid down in a manner convenient for cleaning, which is necessary only once before and after the season, if they are given sufficient fall. Before the mud tanks and the filters there is a channel or pipe *h* leading to the tank *e*, and provided with the necessary valves, so that the waters may be run off direct at the end of the season, or for repairs.

2. Waters from the Bone-Black Treatment, and from Washing Filters and Cloths.—The purification of these waters offers great difficulties, and the temperature in the waste-pipes varies from 20–60° C.; the quantity and variability of the suspended or dissolved impurities are very considerable. These impurities are organic matters, albumin, alkalis, and alkaline compounds, salts, acids, products of fermentation, etc. A large number of experiments have been required to find a universal means for removing these average impurities. The author has used turf coke, which is cheap and best obtained on the large scale by burning in heaps, with the best results for filling the filters d_1 and d_2. It was found that, by reason of the great porosity and consequent absorptive properties of this material, every trace of sugar, salts, fæcal matter, etc., was removed from the waste waters which passed through. The mineral constituents of the turf coke—iron, gypsum, lime—give it even to some extent a disinfecting action; and there is a considerable advantage in that the absorbed matter loses none of its fertilising value for plants, so that the turf coke, together with these absorbed matters, forms an addition to soil which is most valuable in every respect. In regard to a complete disinfection of these waste waters, according to the author's experiments, only the compound of acid magnesium phosphate with basic iron salt—the double salt of magnesia and iron already recommended by Blanchard and Chateau—has been found satisfactory. By this compound the whole of the nitrogen is fixed, with formation of ammonium magnesium phosphate; the other organic matters are also precipitated, the sulphur compounds, sulphuretted hydrogen, etc., by the basic iron salt, so that the water is so far prepared for complete purification that, after passing the settling-tank, it only requires filtering through turf coke.

A similar precipitant to Blanchard's—magnesium sulphate, calcium phosphate, and ferric phosphate—has also, according to the author, been made for several years by Frank, of Stassfurt, and sold at the very low price of 2s. 6d. per cwt. This preparation is used for the purification under consideration in the following manner: 100 parts are stirred with 66 parts of ordinary hydrochloric acid in a tank or tub and allowed to stand two to three days, 300–400 parts of water are then added. The vessel *i* (Figs. 21 and 22) is filled

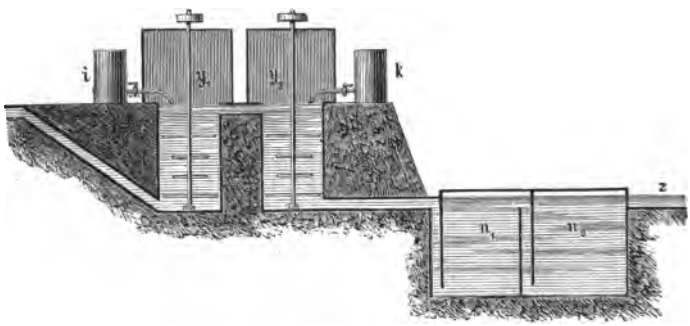

FIG. 21.

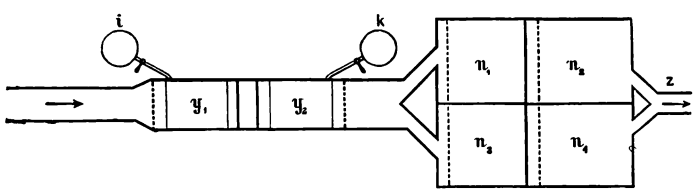

FIG. 22.

with this mixture; as much as is required is run through the exit pipe into the waste water contained in the first section of the apparatus *y*, and well mixed by means of the stirrer. Milk of lime from the second vessel *k* is run into the second section of the apparatus, which is also provided with a stirrer, in sufficient quantity to make the liquid alkaline, so that any free phosphoric acid present may be recovered as tribasic phosphate of lime. The settling of the precipitate, which is generally considerable, takes place in the sections of the settling-tank n_1–n_4, which are connected together two and

two and used in turn. According to the position of the valves, the liquid flows either into the tank n_1 and thence runs over into n_2, or into n_3 and thence to n_4. The clarified water then goes through the drain z to the settling-tanks a_1, b_1, or a_2, b_2 (Figs. 18–20), is mixed with the contents of these tanks, enters into reaction with them, and then runs away for the final purification in the filters c, d_1, d_2.

In regard to the construction of this purifying plant, the tanks y_1 and y_2 are built of wooden planks or bricks; they are about 63 cm. (2 ft.) wide and 95 cm. (3 ft.) high, with two divisions, each 63 cm. (2 ft.) long (round or rectangular). The stirrers are driven in the most convenient manner from the nearest shafting. The liquor is fed in at the bottom and runs away at the top; in the hinder wall of each division is a valve just above the bottom for running off the contents. The vessels i and k are of wood or iron, 79 cm. (2 ft. 7 in.) wide and 95 cm. (3 ft.) high. The settling-tanks n_1–n_4 are of brickwork, open or covered, and connected two and two in such a manner that the water runs over from one into the other; each tank is about 1·3 metres (4 ft. 3 in.) square and 1·6 metres (5 ft. 6 in.) deep.

The precipitate collected in the tanks n_1–n_4 possesses great fertilising value; it has been found by several analyses to contain ammonium magnesium phosphate equivalent to 28 per cent. of phosphoric acid and 10 per cent. of ammonia, also varying quantities of readily soluble basic phosphate of lime, the sulphur compounds combined by the iron salt, and various nitrogenous and organic substances. Together with the deposits in the settling-tanks and the contents of the filters, it produces an excellent fertiliser, the value of which covers all expenses.

3. The Water of Condensation and the Condensed Water from the Evaporation of the Juice.—The temperature of this water varies in the waste-pipe from 30–60° C.; it is contaminated by small quantities of volatile organic matter, ammonia, juice, also fats, fatty acids, etc. Since, as a rule, well-water, containing more or less lime, is used at the lowest possible temperature for condensing purposes, and the lime is partially separated owing to the heating by the

vapours from the juice, this waste water is in this respect more pure than the original water. A portion of this large quantity of water is used for preparing the lime; it may also be used without hesitation for feeding the boilers, and, if necessary, for treating the bone black, also, in very frosty weather, for washing the beet. The remainder, in case the quantity is considerable, must be cooled as much as possible by a suitable simple cooling-tower or terrace cooler, in which the heat should be regained, or by one of the surface coolers used in breweries; the water is then to be completely purified by the method of section 1 or 2, according to the locality. If the excess of water is not great, it will require no separate cooling.

Napravil, in order to recover the fertilising matters and to purify the waste waters, allows milk of lime to run in continuously (Kohlrausch, *Organ für Rübenzücken-Industrie*; Fischer, *Verwerthung der städtischen und Industrie-Abfallstoffe*). The precipitate obtained settles in large flocks, whilst the water runs away quite clear. In 119 working days 3053 cwt. of lime, at a cost of 1005 fl. 95 kr. (£91, 12s.), were used; the cost in wages was 190 fl. 89 kr. (£17, 7s.); 26,228 cwt. of slime were obtained. The best method of removing and utilising these waste waters is to employ them for irrigation.

The **separating or saturation mud** is the most abundant residue produced in sugar-works; several factories obtain in one season 15,000,000–20,000,000 kilos. (15,000–20,000 tons). This residue frequently forms great heaps near the works, which, by reason of the decomposition which sets in, give rise to many inconveniences and complaints in the neighbourhood, and even damage the manufacture itself. This mud has only found one use on the large scale—as a fertiliser; yet it is rather harmful to calcareous soils, since it only contains 0·04 part of phosphoric acid and 0·35 part of nitrogen to 35 parts of lime. The author supposed that by carbonisation a black substance could be obtained, which would find various uses (*Journ. des Fabr. de Sucre*, 20, No. 36). The heating must be so conducted that the organic matter leaves behind carbon after its decomposition. This carbon has the advantage

of being in a very fine state, similar to a true chemical precipitate, so that the material, when squeezed between the fingers, feels like talc or soap. If it is attempted to carbonise the mass in large crucibles, the portion in the middle is not sufficiently heated, whilst the portions at the sides are overheated; also at temperatures higher than a dark red heat the carbonate of lime is decomposed, and the carbon is at the same time oxidised, so that only white lime remains.

It is therefore necessary to use another apparatus; the rotatory furnace of Casalonga is the best. This consists of a revolving cast-iron cylinder, which has a hollow axis, through which the tar and gas can escape. The cylinder makes only a few revolutions per minute. The dried mud is fed in at one end, and escapes at the other by a suitable arrangement after it has been carbonised. The substance obtained consists of an impalpable powder and of small harder particles, which are easily crushed. It is thoroughly powdered and may then be used as a black pigment. Its composition is as follows:—

Calcium carbonate	78·500
Phosphoric acid	0·950
Ammonia	0·168
Ferric oxide and alumina	6·509
Silica	3·205
Carbon	7·500
Undetermined	3·168
	100·000

The percentage of carbon varies according to the composition of the mud—in particular, according to the quantity of sugar it contains. The latter generally soon disappears owing to the fermentation which arises; it is therefore well to add to the mud before carbonisation about 10 per cent. of waste molasses. The shade of the black is more or less good according to the quantity of this addition.

The volatile products evolved in the carbonisation deposit ammonia-water and tar in the condensers, whilst the gas is drawn off into the fires, where it burns with the production of much heat. The tar may also be burnt, if a Deville

furnace is used. The termination of the process is readily recognised by the blue flame which appears when the decomposition begins to yield carbon monoxide. The condensed water contains much ammonium carbonate, ammonia, and other nitrogenous compounds, including pyridine and picoline. In order to separate these products from one another, the liquid is distilled with caustic soda solution, the distillate saturated with hydrochloric acid, and evaporated, when crystalline ammonium chloride is obtained. The mother liquor is precipitated by alcohol, which retains in solution the hydrochlorides of the bases containing carbon, which are then set free by potash.

Further investigations of these products have been made by Etard (*Zeits. d. Ver. f. Rübenzucker-Industrie*, 16, p. 916), they are similar to those obtained by Vincent from distillers' refuse. The principal product is naturally the black, which is suitable for use in size, oil paints, and printing inks. The cost is only 10 francs per 100 kilos. (4s. per cwt.).

Skene, of Breslau, has given an account of the arrangements he employs for the waste waters (*Zeits. d. Ver. f. Rübenzucker-Industrie;* Post, *Zeits. f. d. chem. Grossgew.*). The whole of the waste water goes on to 17·5 hectares (43·2 acres) of land, surrounded by banks; here the water cools; it is then drained off, and again filtered by passing through a meadow of 3·25 hectares (8 acres). The drainage is laid down in the ordinary system and is cut off by a valve, which is not opened until all the conduits have been pumped full. The irrigation works are so large, that the water is only run off once in the season.

Purification of the Alumina which has been used for Clarification in the Manufacture of Sugar, in order to recover the Matter it contains.—The brothers Löwig have patented four processes for this purpose (Post, *Zeits. f. d. chem. Grossgew.*):—

1. The used alumina is mixed with one equivalent of lime and ignited. Calcium aluminate is formed, the organic matter first leaves carbon or burns entirely on further heating. Ammonia, which can be recovered, is formed from the nitrogen in the organic matter.

2. The alumina is heated with magnesium chloride; magnesium aluminate is then formed, whilst the organic matter is destroyed by the hydrochloric acid produced, which can also be condensed and used later to dissolve the magnesium aluminate. The aluminates obtained in these processes (1 and 2) are converted into alumina by known methods.

3. The alumina is dissolved in the least quantity of hydrochloric acid, and the solution filtered from insoluble organic matter, *e.g.* albumin from plant juices containing sugar. Many of the organic substances which remain in solution can then be precipitated by the addition of alumina, or by the precipitation of a portion of the dissolved alumina, and then filtered off. The aluminium chloride may also be ignited, and the organic compounds destroyed by the hydrochloric acid which is formed; but care must be taken that too violent heating does not produce insoluble alumina.

4. The impure alumina may further be dissolved in caustic alkalis and thus freed, for example, from ferric oxide; then, if necessary, it may be purified by igniting the evaporated solution, and finally again be precipitated as the aluminate of an alkaline earth. The albuminous matters obtained in method 3 may be used as fertilisers or in the manufacture of ammonia.

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